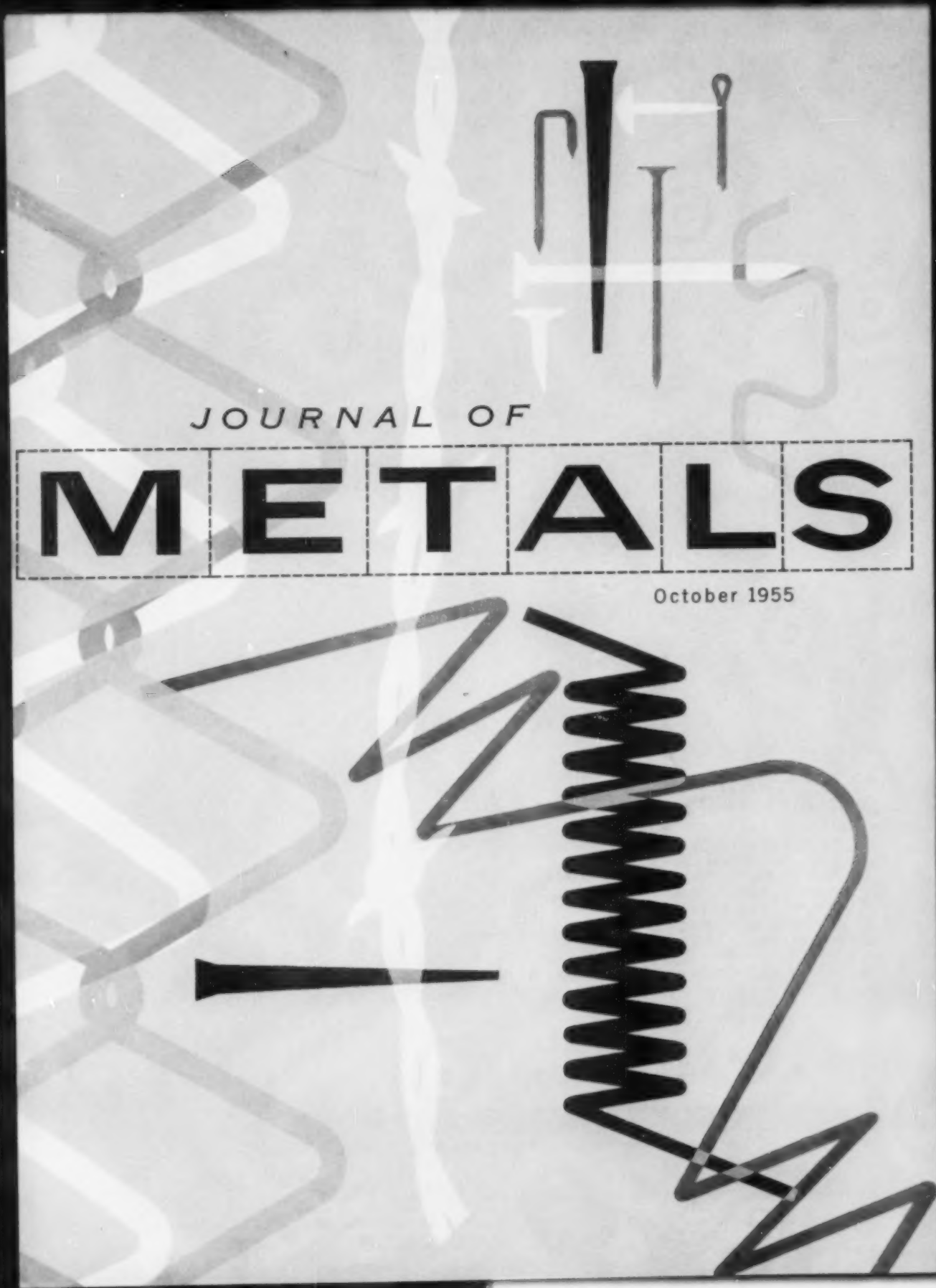

JOURNAL OF

METALS

October 1955

The New DUPLEX
AJAXOMATIC

AN INTEGRATED MELTING AND AUTOMATIC POURING UNIT FOR THE PRODUCTION OF ALUMINUM DIE CASTINGS

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JOURNAL OF

METALS

Published to provide a continuing, authoritative, and up-to-date record of technological, engineering, and economic progress in all branches of the metals industry by the
Metals Branch
American Institute of Mining and Metallurgical Engineers, Inc.
29 West 39th Street, New York 18

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Journal of Metals
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VOL. 7, NO. 10 OCTOBER, 1955

COVER

Cover by Harry Brocke illustrates the variety of metal shapes and designs.

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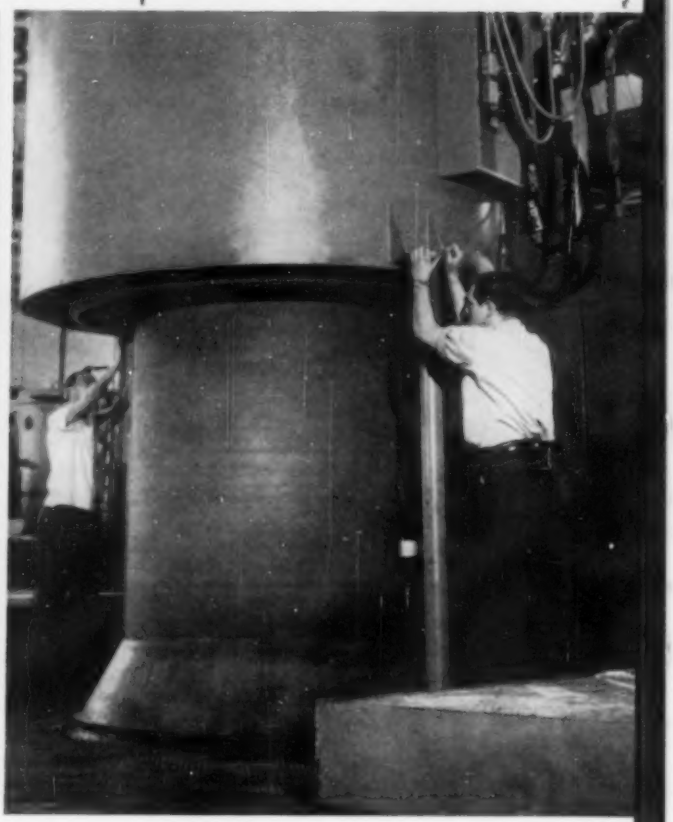
Stokes vacuum brazing furnace at Pratt & Whitney Aircraft offers complete accessibility to work area and simplicity in material handling. These three simple operations are involved: (1) placing of work (2) covering work with retort and (3) lowering the furnace shell.

Pratt & Whitney



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Stokes manufactures a complete line of vacuum pumping equipment. This includes mechanical vacuum pumps, diffusion and booster pumps, vacuum valves and gages, and complete vacuum instrumentation. In engineered high vacuum equipment, Stokes builds vacuum metallizers, vacuum furnaces and other vacuum processing equipment.

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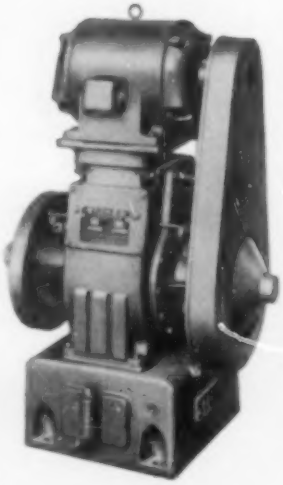

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Aircraft Uses Stokes Furnace to Investigate Vacuum-Brazing of Jet Engine Components

Versatile new furnace design also can be used for heat-treating and outgassing at high vacuum or under controlled atmospheres.

A logical development of vacuum metallurgy, high-vacuum brazing, presently being investigated by numerous firms throughout the country, promises to produce superior products more economically than brazing methods used heretofore. Tests of vacuum brazed components indicate the greater strength, ductility and uniformity of assemblies produced by this method.

Typical of firms currently carrying on investigations in this field is Pratt & Whitney Aircraft, East Hartford, Conn. The firm is using a Stokes furnace in experimental work on the application of vacuum brazing to the production of jet engine components.

Stokes' furnace design for vacuum brazing is practical . . takes into account the problems of large-scale production. The Stokes dual-chamber design, with a smooth-contour retort completely enclosing the work, permits faster vacuum pumping cycles; provides complete accessibility of the work area and a large, uniform temperature zone.

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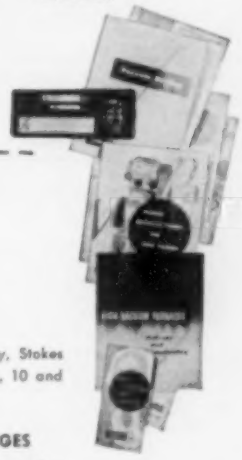
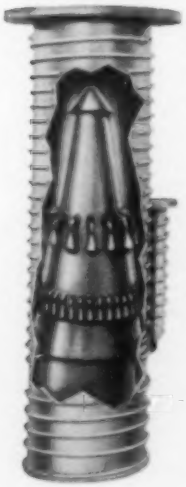
SEND FOR TECHNICAL LITERATURE:

- Microvac Pumps—Catalog 750
- Diffusion and Booster Pump Specification sheets and performance curves
- The Story of the Ring-Jet Pump
- Complete Vacuum Processing Systems—Catalog 730
- How to Care for Your Vacuum Pump—Booklet 755
- Vacuum Impregnation—Catalog 760
- Vacuum Drying—Catalog 720
- Vacuum Furnaces—Catalog 790
- Vacuum Metallizing—Catalog 780
- Vacuum Calculator Slide Rule



VACUUM EQUIPMENT



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Metallurgist (woman), M.S. degree, 16 years research and control experience in light and electron metallography, powder metallurgy, corrosion, on steels, copper, tungsten, titanium; desires responsible position, preferably in research within commuting distance of Bridgeport area, Conn. M-144.

Metallurgical Engineer, M.S., 31. Five years experience research, development and process control. Includes metalworking steel, nonferrous and powder metallurgy. Graduate studies. Desires position with future leading into management with progressive organization. Location immaterial. M-145.

POSITIONS OPEN

Engineers. (a) Research process metallurgist, master's or doctor's degree in metallurgy, with up to five years experience, for research and development in nonferrous extractive metallurgy in research dept. of nonferrous mining and smelting company. Pyrometallurgy, roasting, smelting, process research for production of nonferrous metals, including titanium. (b) Research physical metallurgist, master's or doctor's degree in physical metallurgy with up to five years experience for research in product development group of research dept. for producer of nonferrous metals and alloys, alloy development, metal finishing, corrosion and electrochemical research. Location, East. W2053.

Engineers. (a) Supervisor of magnetic materials in research and development dept. Must have Ph.D. in physics or metallurgy and minimum of five years experience in research or development of magnetic, Alnico and Ferrites, precision casting, powdered metallurgy. Salary open. (b) Silicon steel research supervisor, with degree in metallurgy or physics, for research and development dept. Must have minimum of five years experience in research and development of steel for the electrical industry. Must be very high type of caliber. Will hire and supervise his own staff. Salary open. Location, western Pennsylvania. W2042.

Engineers. (a) Associate metallurgists, B.S. in metallurgy or physics, for research and development dept. Must have minimum of four years experience in research and metals and have ability to write reports and comprehend metallurgical processes. Salary open. (b) Research metallurgists, male or female, for research dept. Must be graduate of recognized technical school and have minimum of five years experience in research and development of metals and alloys. Must be able to outline and conduct research and development program, expand use of the present steels and improve steels.

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—reactor technology
—high-temperature and mechanical metallurgy
—minerals processing
—metallurgical economics

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The Personnel Manager
BATTELLE MEMORIAL INSTITUTE
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Columbus 1, Ohio

Salary open. Location, western Pennsylvania. W2041.

Metallurgist with experience in X-ray diffraction and X-ray fluorescent analysis studies and their application to metallurgical problems. Salary open. Location, Northwest. W2028.

Metallurgist, 26 to 40, metallurgical graduate, with metallography, inspection, extrusion and production experience in wire or tube mills. Salary, $6500 to $8500 a year. Location, Ohio. W2016.

Research Engineers. (a) Research engineer or research metallurgist, Sc.D. or M.S. in mineral engineering or metallurgical engineering, to conduct research leading to better methods of mineral recovery. Two to three years experience as a research engineer is desirable. Must have demonstrated ability for initiating and directing experimental work in fields of mineral dressing or extractive metallurgy. (b) Research engineers, Ph.D. or Sc.D. in chemical engineering or extractive metallurgy for duties focused on the technology of extremely pure metal compounds and their subsequent reduction to metals. Will have responsibility for bench scale to pilot scale work on unit operations such as leaching, chlorination, distillation, melting, high temperature furnace work, solvent extraction and ion exchange. Ability in preliminary design is desirable. Location, New York State. W1998.

Physical Chemists. (a) One with Ph.D. in physical chemistry or metallurgy for investigation of the physical chemistry of reactions occurring during melting and refining of metals in connection with the design of new alloys based principally on heavy, nonferrous metals and the newer metals such as titanium. Research experience or dissertation in molten metal equilibria preferred. (b) One with Ph.D. in physical chemistry or physical metallurgy for investigation of reactions of solid metal with gas at elevated temperatures. Emphasis will be on the oxidation of metals. Apply existing oxidation theory in an attempt to improve the oxidation resistance of present high temperature alloys and to develop oxidation resistant alloys based on the newer metals such as titanium. Research experience or dissertation on solid metal gas reactions preferred. Location, New York State. W1997.

Metallurgists. (a) Physical metallurgist, M.S. or Ph.D. or equivalent in physical metallurgy, for investigation of kinetics of solid state reactions leading to the design of new alloys for high temperature applications as well as alloys based on reactive metals such as titanium. Must be capable of originating and directing research programs in alloy design. Research experience in field of alloy design desirable. (b) Solid state physicist or metallurgist, Ph.D. in solid state physics or metallurgy, to conduct fundamental research in the field of magnetic materials. Work should lead to design of new alloys for improved permanent magnets. Substantial dissertation research experience on magnetic properties of materials will be adequate background. Location, New York State. W1996.

Metallurgist, with at least a B.S. degree with major in metallurgy from a first class engineering college, and at least two years actual milling experience. Salary, $4800 to $6000 a year, Canadian currency. Location, Canada. W1986S.

Sales Engineer with metallurgical engineering training and melting experience, to call on foundries and furnaces. Salary, $5000 to $6000 a year plus bonus. Territory, East; headquarters, New Jersey. W1980.

Metallurgical Engineer, young, to act as assistant to chief metallurgist, for a fabricator of sintered metal parts on a custom molding basis. Salary open. Location, Connecticut. W1978.

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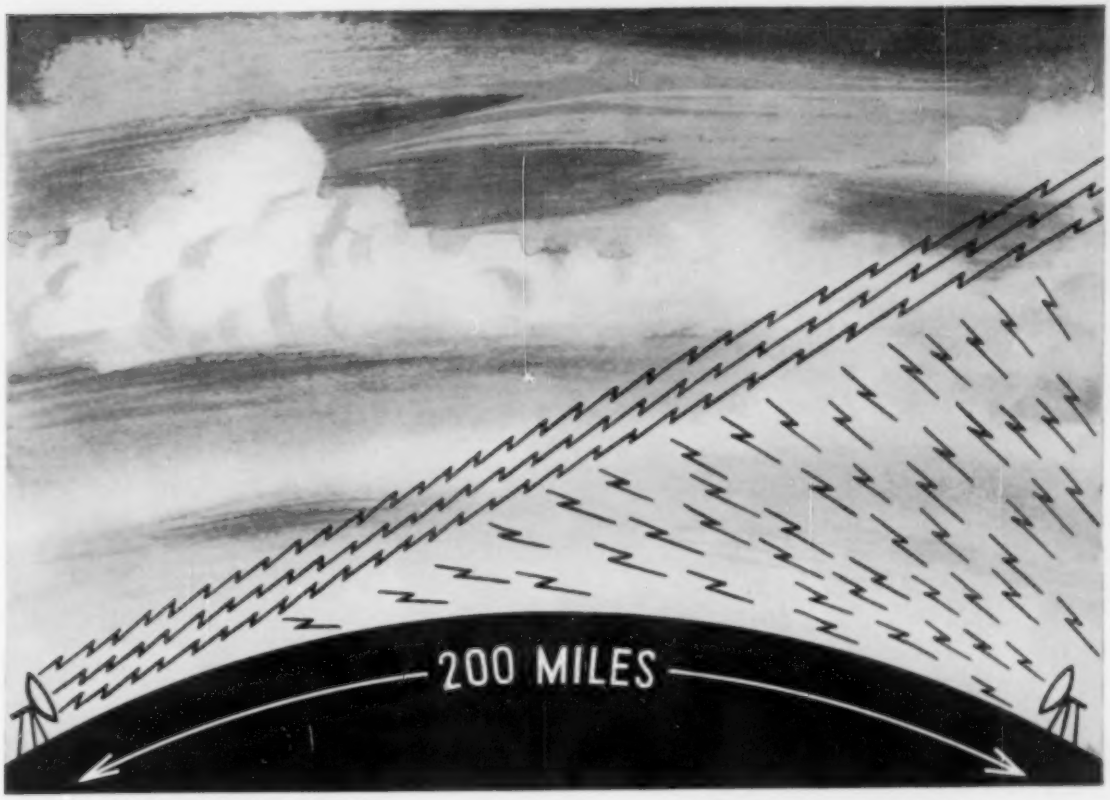
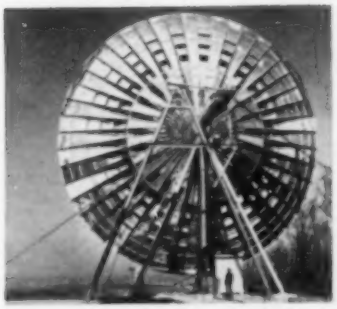


Highly schematic drawing illustrates the possible distribution of energy in ultra-high-frequency "over-the-horizon" transmission. The effect is similar to that of a powerful searchlight whose beam points into the sky. Light can be seen miles away from behind a hill even when the searchlight lens is invisible.

Something new on the telephone horizon



This experimental 60-foot antenna (rear view) photographed at Bell Laboratories in Holmdel, New Jersey, is designed for study of "over-the-horizon" phenomena.

Telephone conversations and television pictures can now travel by ultra-high-frequency radio waves far beyond the horizon. This was recently demonstrated by Bell Telephone Laboratories and Massachusetts Institute of Technology scientists using "over-the-horizon" wave propagation, an important recent development in the radio transmission field.

This technique makes possible 200-mile spans between stations, instead of the 30-mile spans used for present line-of-sight transmission. It opens the way to ultra-high frequencies across water or over rugged terrain, where relay stations would be difficult to build.

In standard microwave line-of-sight transmission, stations are so spaced that the main beam can be used. But now, with huge 60-foot antennas, and much higher power, some signals drop off this main beam as it shoots off into space. These signals reach distant points beyond the horizon after reflection or scattering by the atmosphere. The greater power and larger antennas of the "over-the-horizon" system permit recapture of some of these signals and make them useful carriers. The system will be a valuable supplement to existing radio relay links.

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— Coming Events —

Oct. 2-5, AIME, MGGD fall meeting and Black Hills regional meeting, Ind. Min. Dv., Alex Johnson Hotel, Rapid City.

Oct. 3-5, National Electronics Conference, Hotel Sherman, Chicago.

Oct. 6-7, AIME, NOHC, Southwestern Section, Kansas City, Mo.

Oct. 6-8, AIME, Rocky Mountain Minerals Conference, Hotel Newhouse, Salt Lake City.

Oct. 7-8, American Ceramic Society, refractories div., Bedford Springs, Pa.

Oct. 9-13, Electrochemical Society, William Penn Hotel, Pittsburgh.

Oct. 11-15, SAE, aeronautic meeting, Hotel Statler, Los Angeles.

Oct. 12-14, Iron and Steel Institute, Scunthorpe, England.

Oct. 13-14, Engineers' Council for Professional Development, annual meeting, King Edward Hotel, Toronto, Canada.

Oct. 14, AIME, NOHC, Eastern Section, all day technical meeting, Warwick Hotel, Philadelphia.

Oct. 14-15, National Society of Professional Engineers, fall meeting, Peabody Hotel, Memphis.

Oct. 17-19, AIME, IMD, fall meeting, Adelphia Hotel, Philadelphia.

Oct. 17-21, National Metal Exposition and Congress, Philadelphia.

Oct. 17-21, National Safety Congress and Exposition, Conrad Hilton, Congress, Morrison, and LaSalle Hotels, Chicago.

Oct. 19-21, Columbia School of Engineering, annual conference, representatives of Combined Plan for a Liberal Education in Engineering, Arden House, Harriman, N. Y.

Oct. 20-21, National Noise Abatement Symposium, Armour Research Foundation, Illinois Institute of Technology, Chicago.

Oct. 24-25, Steel Founders Society of America, fall meeting, Greenbrier, White Sulphur Springs.

Oct. 24-26, American Standards Assn., national conference, Sheraton Park Hotel, Washington, D. C.

Oct. 24-29, Societe Francaise de Metallurgie, autumn metallurgical meeting, Paris, France.

Oct. 26-28, National Industrial Conference Board, atomic energy in industry, Waldorf-Astoria Hotel, New York.

Oct. 27-28, National Conference On Industrial Hydraulics, annual meeting, La Salle Hotel, Chicago.

Oct. 27-29, AIME, Ind. Min. Div., fall meeting, Hotel Charlotte, Charlotte.

Oct. 27-29, Oak Ridge Institute of Nuclear Studies, rare earths in biochemical and medical research conference, Oak Ridge.

Oct. 31-Nov. 2, National Lubricating Grease Institute, annual meeting, Edgewater Beach Hotel, Chicago.

Nov. 1-3, Investment Casting Institute, annual meeting, Detroit.

Nov. 1-5, World Symposium on Applied Solar Energy, Westward-Ho, Phoenix.

Nov. 2, SAE, The Chase, St. Louis.

Nov. 4, AIME, Pittsburgh Local Section, off-the-record meeting, William Penn Hotel, Pittsburgh.

Nov. 7-8, Western Governors Mineral Policies Conference, Sacramento.

Nov. 7-9, Eastern Joint Computer Conference, Hotel Statler, Boston, Mass.

Nov. 9, Stanford Research Institute, associates' day, Stanford, Calif.

Nov. 9-11, Industrial Management Society, time and motion study and management clinic, Sherman Hotel, Chicago.

Jan. 19, 1956, AIME, NOHC, Western Section, Rodger Young Auditorium, 936 W. Washington Blvd., Los Angeles.

Feb. 20-23, AIME, annual meeting, Statler and New Yorker Hotels, New York.

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Books for Engineers

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The Handbook of Microscopy in Technology, edited by Hugo Freund. *E. Leitz, Inc.*, 8 vol., $30 to $40 each, 1955—Available only in German, these volumes give a complete coverage of microscopy in many technological fields including minerals, ores and metals, coal and coke, wood, paper, textiles, chemical, etc. Profusely illustrated, the volumes contain contributions from well over 100 of Europe's outstanding scientists in the various fields. The collection should be of considerable assistance to chemists, metallurgists, and others in industrial research laboratories.

Dictionary of Mechanical Engineering Terms, by J. G. Horner, revised by Staton Abbey. *Philosophical Library, Inc.*, 7th edition, $6.50, 417 pp., 1955—This volume is a comprehensive dictionary of the general and traditional terms used by draftsmen, patternmakers, molders, smiths, boilermakers, fitters, turners, erectors, and engineering storekeepers. The appendix has been completely revised to include terms which have come into use during recent years.

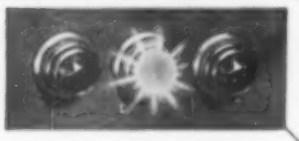
Magnetic Materials in the Electrical Industry, by P. R. Bardell. *Philosophical Library, Inc.*, $10.00, 288 pp., 1955—This book is intended for students in physics and electrical engineering and for physicists and engineers in industry. It bridges the gap between an academic study of the properties of magnetic materials and the limited treatment of the subject possible in most textbooks for engineers. One of the aims is to link the properties of materials with their applications. A glossary of terms and units is included.

Tungsten, by K. C. Li and Chung Yu Wang. *Reinhold Publishing Corp.*, third edition, $14.00, 506 pp., 1955—Every aspect of the history, geology, ore dressing, metallurgy, chemistry, analysis, industrial applications, substitutes, and economics of tungsten is covered in detail. Ample literature references provide helpful guidance for further research.

LaPlace Transforms for Electrical Engineers, by B. J. Starkey. *Philosophical Library, Inc.*, $10.00, 279 pp., 1955—A physical rather than a purely mathematical vocabulary is used in an attempt to attain the utmost simplicity. The approach throughout is from analytical methods already well known to engineers. The work is intended to be a general introduction to a rather large subject. The LaPlace transformation theory is extremely useful in providing quick solutions to a great range of engineering and physical problems and often saves laborious calculation by other methods.

Electro-Magnetic Machines, by R. Langlois-Berthelot. *Philosophical Library, Inc.*, $15.00, 535 pp., 1955—In this English edition, translated and revised in collaboration with H. M. Clarke, the various kinds of electrical machines are related to each other with the recognition that they are of one family with a common basis of experience. Fundamental principles rather than practices are stressed in this work. Students, teachers, technicians, industrialists, and anyone concerned with electrical plants will find the book of interest.

The Metallurgy of Zirconium (National Nuclear Energy Series), edited by B. Lustman and F. Kerze, Jr., first edition, *McGraw-Hill Book Co., Inc.*, $10.00, 776 pp., 1955—Presents a critical review by more than 50 contributors of the many aspects of zirconium technology. Occurrence, reduction, refining, properties, and uses are covered in detail.



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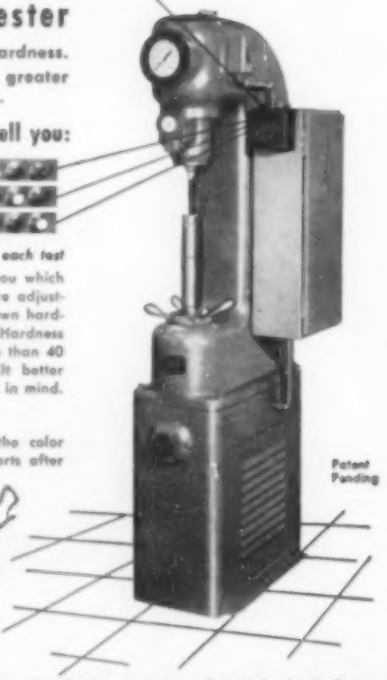

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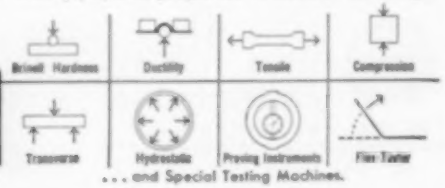

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United States Steel's Edgar Thomson Works in Braddock, Pennsylvania, needed new and heftier crane equipment to handle larger ladles. One of the requirements was four new crane trolleys which were to operate on the same bridges, if possible, and in exactly the same clearances as before.

The trolleys had to be stronger but no larger than before; so ordinary steel wouldn't do. Needed, was a steel of *exceptional strength* which could be *welded easily*. Lots of alloy steels could have met the strength requirements; but USS "T-1" Steel was the *only* steel that could supply not only the required strength, but good weldability as well.

"T-1" Steel plate—in ¾" and 1" thicknesses—was used in box girders and lateral stiffener plates of the trolleys. This change increased the crane capacity from 50 to 85 tons with no increase in size.

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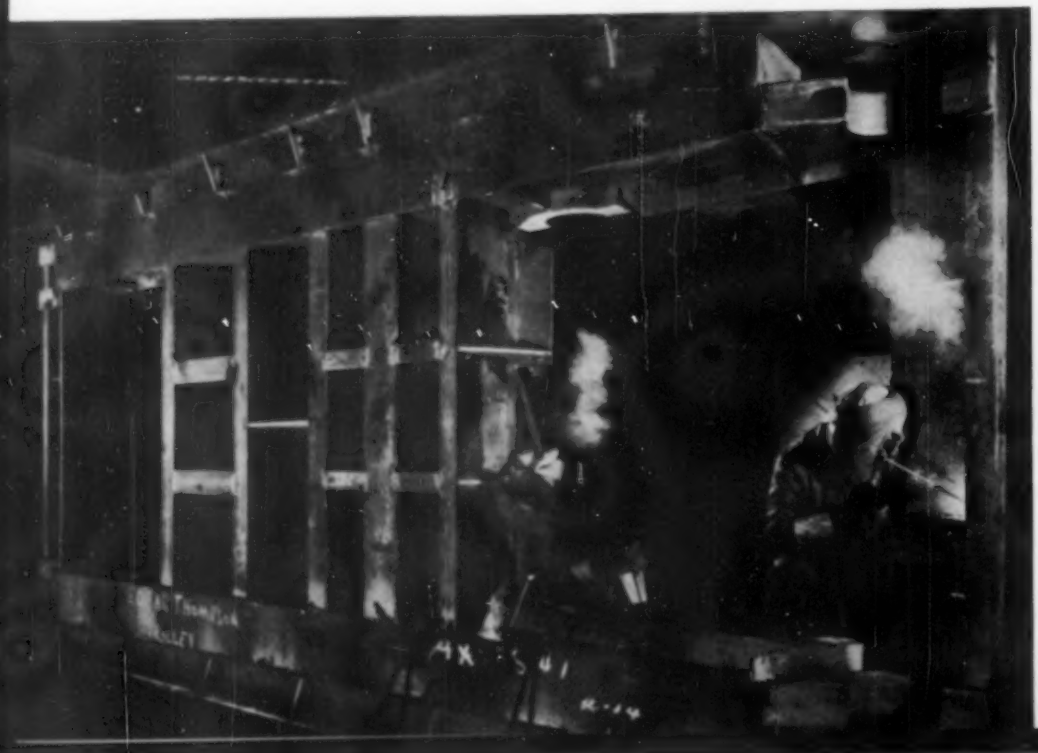

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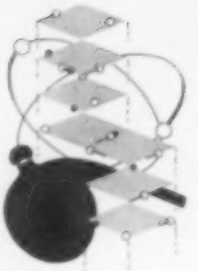

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Industrial Notes

• *Salem-Brosius, Inc., Carnegie, Pa., was appointed agent for the sale and licensing of an ultra-high temperature furnace and the processes of Metals Chlorides Corp. of Middleport, N. Y. The new furnace operates at temperatures in excess of 4000°F and is designed for continuous tonnage halogenation of oxides, ores, and carbides at temperatures near to, or above, the vapor stage temperature of most metals and metallic compounds.*

• **A booklet** *How Zinc Controls Corrosion* **presents information for management, maintenance engineers, architects and anyone concerned with minimizing corrosion and maintenance costs. The booklet can be obtained from: American Zinc Institute, 60 E. 42nd St., New York 17.**

• A quenching oil to give extremely fast cooling rates in conventional quench set-ups has been developed by the Shell Oil Co. Named Shell Voluta Oil 23, the oil has unusually high oxidation stability and causes very little smoke and flame during normal hardening operations. Flash point, fire point, and sp gr are 380°, 425°F, and 29.9° API, respectively.

• A compilation of available technical and fabricating data on arc cast molybdenum is presented in a 72-p. booklet published by Climax Molybdenum Co. It is designed to provide information on the high temperature applications of molybdenum with a complete picture of where the metal stands today and possible uses in the near future. A copy of *Arc-Cast Molybdenum and its Alloys* can be obtained by writing: Director of Technical Information, Climax Molybdenum Co., 500-5th Ave., New York 36.

• **A 12,000 ton horizontal extrusion press was built by the Lombard Corp., Youngstown, Ohio. The Lombard press, the largest American made extrusion press, will contribute to removing restrictions previously imposed upon designers of large military aircraft in regard to size, weight, and length of extrusions available for plane components. It was built for the Air Force as part of its Heavy Press Program.**

• *The U. S. Testing Co., Hoboken, N. J., has published* Selected Scientific and Engineering Tables and Data. *Issued in commemoration of the company's 75th anniversary, the 112-p. paper bound volume contains selected chemical and physical, engineering, plastics, bacteriological, leather, psychometric, and textile tables and charts. A copy can be obtained by writing on company letterhead to: H. M. Block, vice president, U. S. Testing Co., Hoboken, N. J.*

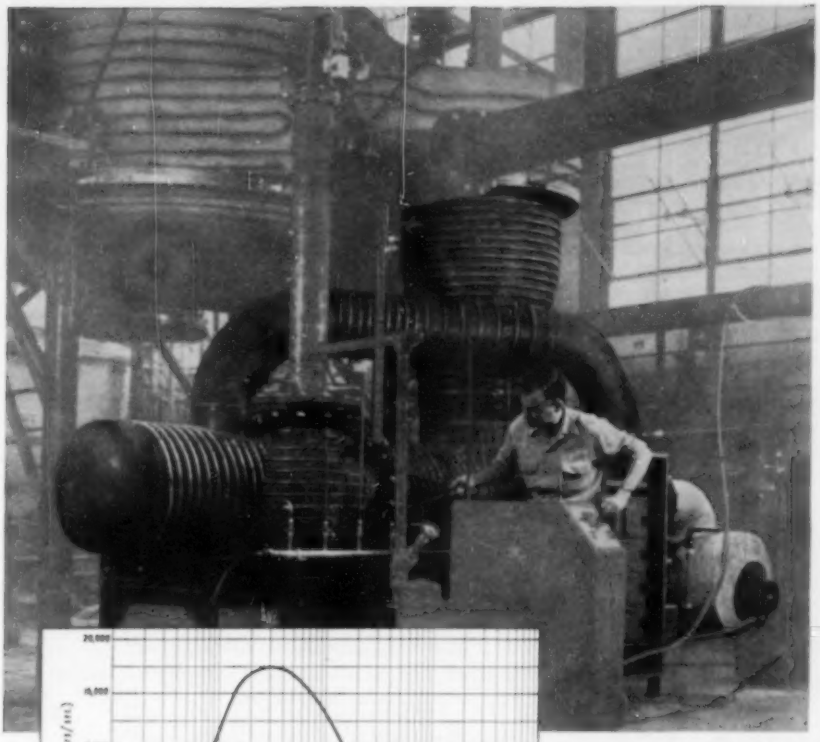
• A 32-p. illustrated guide to specialty steels was issued by The Carpenter Steel Co. It briefly summarizes the company's specialty product line covering tool and die steels, stainless steels, silicon and high nickel electrical alloys, special purpose alloy steels, and heat resisting and super alloy steels. Corollary information is also given on the quality control necessary in the manufacture of specialty steels. A copy can be obtained from: Carpenter Steel Co., 364 W. Bern St., Reading, Pa.

• *Development of a new metallurgical microscope was announced by Bausch & Lomb Optical Co., Rochester, N. Y. New features include an oversized focusing stage with interchangeable stage plates which adapt it quickly for work with either transparent or opaque specimens, a fine adjustment focusing knob located at table level to permit operators to work with their arms in a relaxed position, and an accessory lens for concentrating external illumination into the microscope for photomicrographic use.*

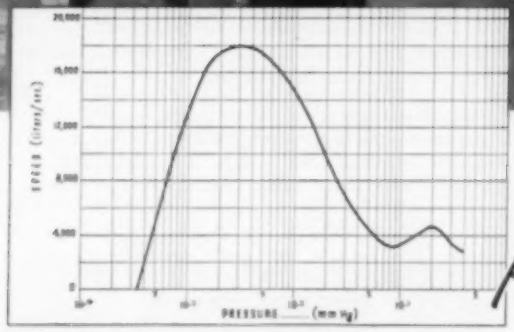
• **Union Carbide Nuclear Co., a division of Union Carbide & Carbon Corp., has been formed to integrate the corporation's diverse activities in the atomic energy field. Kenneth Rush has been appointed president of the new company. An important objective will be to carry on the large-scale research and development activities leading to increasingly important participation by the corporation in the industrial applications of atomic energy.**

• A new graphite brick that has shown a 25 pct saving in cupola wells is the product of Mexico Refractories Co., Mexico, Mo. Giving up to 31 days service in a cupola well carrying 350 to 450 tons per day in tests so far, the Helspot brick has shown a substantial saving in pouring cost for cupola wells. Helspot brick are characterized by low porosity, negligible shrinkage, and good uniform structure.

• *Reynolds Metals Co. has issued the 1955 edition of* Aluminum Bridge Railings Manual. *The 52-p., 8½x11 in. manual contains 117 illustrations and 23 tables. Subjects treated in the manual are: all-aluminum lighting standards, design criteria, detailed analysis of typical bridge railings, and joints, endings and post settings. Another important feature is that reference and mill die numbers are listed along with all extruded shapes for ready identification. The manual will be sent without charge upon request to: Desk PR, Reynolds Metals Co., 2500 S. Third St., Louisville 1, Ky.*



The type KS-16,000 diffusion-ejector pump undergoing helium-leak check on a CVC one-thousand pound high-vacuum melting and casting furnace installation.



Type KS-16,000's performance curve.

Pressure falls fast

with this new CVC high-vacuum pump

Removing *1,000,000 micron-liters of gas a second* from a furnace chamber—that's the task of *CVC's* new diffusion-ejector pump, type KS-16,000.

1,000,000 micron-liters a second, or 12.5 pounds of air per hour, means that this *single* pump can exhaust a one-thousand pound melting and casting furnace smoothly and swiftly and maintain the desired operating pressure throughout outgassing periods.

1,000,000 micron-liters a second also means that *CVC* is able to supply efficient pumping for the tremendous gas loads of the five-thousand and ten-thousand pound high-vacuum furnaces metallurgists plan for the near future.

This high-speed, high-vacuum pump is the latest in a series of advances made in the *CVC* laboratories—advances helping to make vacuum metallurgy an accepted and profitable production process.

Other features of the type KS-16,000:

1. Speed for air is 18,000 liters a second at 4 microns Hg (see chart). Speed for hydrogen is 42,000 liters a second at 6 microns Hg.
2. Ultimate pressure is 4×10^{-4}mm Hg.
3. Limiting forepressure is 10mm Hg.
4. Clearance between jets and pump casing is extra wide and unobstructed. *Dust particles found in various metallurgical processes will not clog jet action.*

For further information about the type KS-16,000 or about the use of high vacuum in metallurgy, write to ***Consolidated Vacuum Corporation, Rochester 3, N. Y.*** (a subsidiary of ***Consolidated Engineering Corporation, Pasadena, California***).

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AN eight lane, half mile long roadway suspended in midair by cables looks like a quote from science fiction. But this in simple words is a suspension bridge. Up in Michigan a new bridge of this type is being built with a main span of 3800 ft, second longest in the world. Five miles in total length, the Straits of Mackinac Bridge will be used to get across the neck of water between Lakes Michigan and Huron which separates northern and southern Michigan.

To get across could have been the slogan of John A. Roebling, the great pioneer of the suspension bridge. Over 100 years ago, Roebling journeyed to Ohio and built the Wheeling Suspension Bridge over the Ohio River. The bridge is still an important east-west link. Plans are now underway for memorial rites to honor the ingenuity of the master bridge builder. The governors of Ohio and West Virginia will head delegations from their respective states.

Roebling's principles, augmented by the latest techniques and experiences of a century, will be applied to the Straits of Mackinac Bridge now under construction. U. S. Steel Corp.'s American Steel & Wire Div. is fabricating the nearly 46,000 miles of wire for the two 8362-ft main cables as well as the 368 vertical suspender ropes that support the weight and stresses of the roadway spans.

Step by step, top quality is a must on bridge wire. Nearly 12,000 tons of special high carbon steel will be drawn to 0.196 in. wire and galvanized. The wires will be spliced into endless strands and laid parallel to one another. The 2-ft diam cross-section of each main cable will consist of over 12,000 individual wires each with a minimum average tensile strength of 225,000 psi. After these mile and a half long 24-in. cables are erected and are near their dead load tension, the compacted wires will be coated with a spirally wrapped wire 0.162 in. in diam.

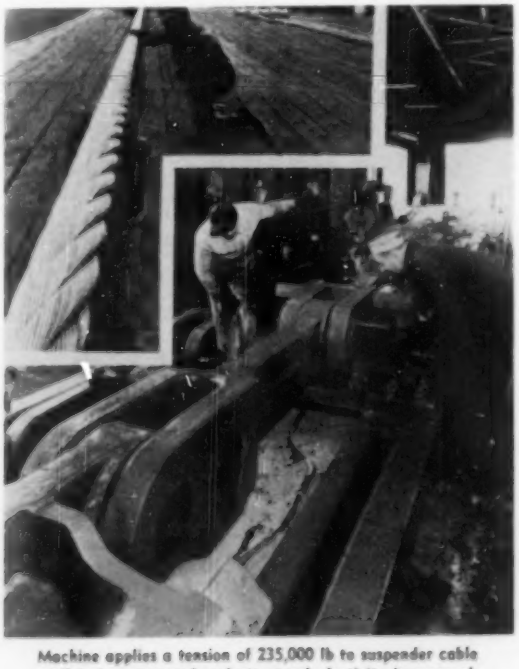
Suspender ropes, 368 of them, hung at 39-ft intervals throughout the length of the bridge will connect the roadway truss to the main cables. These ropes will be 2¼ in. in diam and will be made up of six strands of 19 wires with a total weight of 9 lb per ft. During the process of fabrication the ropes are held at a tension of over 115 tons. This operation is called prestressing and removes the stretch from the rope and allows a means for accurate measurement of lengths.

The metallurgist can take special pride in crossing such a bridge. Under his direction the blast furnace and open hearth have refined the metal and the steel mill has fabricated the components. In the laboratories, metallurgists by their skill and research give the bridge designer the specifications and materials to create these spectacular and beautiful engineering triumphants.

The main span of the Wheeling Suspension Bridge is 1010 ft long, the longest in the world when completed a century ago. The bridge is 28 ft wide from suspender to suspender and it is an average of 91½ ft above low water level.

◆◆◆◆◆◆

THERE'S a new trend in American philanthropy —more and more large corporations are establishing new funds to help colleges and universities. Estimates based on U. S. Treasury figures are that corporate gifts to higher education have increased from $50 million in 1950 to almost twice that amount today. A large part of this increase, of course, reflects the general increase in all corporate gifts. But the Council for Financial Aid to Educa-



Machine applies a tension of 235,000 lb to suspender cable until it is compacted and structural elasticity is removed. Insert is a closeup of a rope under tension in the prestressing beds at American Steel & Wire Div. plant.

tion, Inc., of New York, believes that higher education's share of the philanthropic dollar probably has been boosted from 20 pct in 1950 to 25 pct today.

Within the past year, several of America's largest firms have instituted great new programs of financial aid to colleges and universities. A steel firm will give $1 million this year; another $1 million will be donated by an electrical firm, while an auto firm has added $2 million to a program already totalling $2.5 million and intended to increase by $2.5 million more, in the next four years. Many more millions will be given by other firms this year and in the immediate future. Significantly, small liberal arts colleges, as well as technical institutions, are sharing generously in these corporate gifts.

Business men generally earnestly support this trend of increased corporate giving to higher education for a very practical reason—actual studies show that business is best where educational standards are highest. Business and education have a reciprocal stewardship; business to support a dynamic system of higher education; education to teach the function of the American economic system, and the need for safeguarding and perfecting it.

◆◆◆◆◆◆

ON Friday, August 26, the General Electric Co. dedicated its new $5 million metals and ceramics building. As part of the expansion program of the research laboratory at the Knolls, the building is further recognition by GE that growth of the company can only be insured by continued investment in scientific research and development. Much of the space in the new building is devoted to small factory equipment to test new materials and processes on a scale approaching industrial conditions.

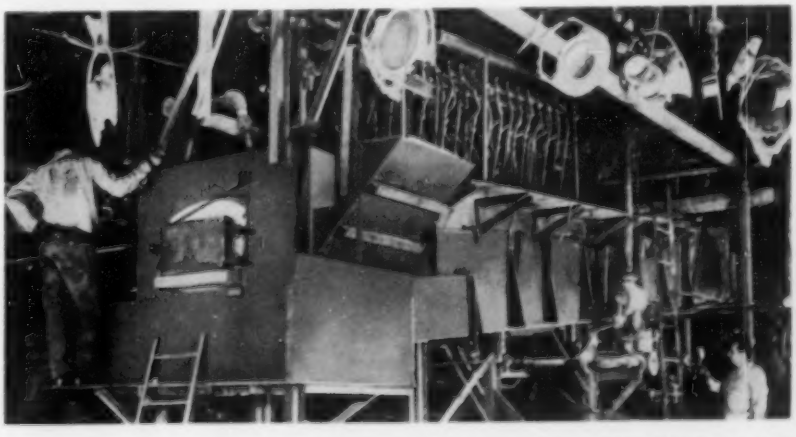
C. G. Suits, vice president and director of research for GE, presided at the dedication ceremonies. Philip D. Reed, chairman of the board, Charles E. Wilson, former president of GE and an employee for 51 years, and J. H. Hollomon, manager of the metallurgy and ceramics research dept., participated in the ceremonies. Donald A. Quarles, Secretary of the Air Force, gave the dedication address.

GE is one of the nation's largest consumers of metals, and the new metals and ceramics building will be the centralized facility to encourage understanding of present materials and processes and to create new ones. According to GE personnel statistics, metallurgists (including metallurgical engineers and those with degrees in the science of metallurgy) constitute roughly 2 pct of the engineers employed by General Electric. At the research laboratory, the employment is 15 pct, representing about 50 metallurgists out of a technical staff of over 300.

Emphasizing the importance of the staff that carries out the research at GE and, elsewhere as well, the dedication ceremonies were preceded by four days of conferences. The first was a two and one half day conference among three dozen selected professors of metallurgy from all over the country and an equal number of industrialists plus GE staff members. The general theme of this conference was industry's needs for metals and metallurgists.

An Education and Manpower Symposium pointed up the fact that in 1950 the U. S. census reported 520,000 professional engineers of which 5 pct were working in metallurgy or mining. In spite of an increasing demand for metallurgists, the postwar peak of engineering graduates of approximately 50,000 has now dropped to about 20,000; and the proportion of metallurgical engineers is also decreasing with a figure of 2.35 for 1954. The shortage of metallurgists with postgraduate degrees has lead GE to increase its four fellowships in this field to eight this fall.

On Wednesday afternoon and Thursday, university professors joined approximately 300 metallurgical engineers and scientists from industry for a Seminar on Melting and Solidification. Emphasized throughout this review of progress to date in the understanding of melting and solidification was the similarity between metals and ceramics. The seminar drew together a large proportion of leading metallurgists, including Sir Charles Goodeve of the British Iron & Steel Research Assn.



Continuous melt aluminum furnace was developed jointly by Monarch Aluminum Mfg. Co. and Selas Corp. of America. Furnace produces 2400 lb per hr of liquid aluminum from ingot for direct pouring into permanent molds.

Drift of Things

ADRIFT would be a more appropriate title for this month's column. During August two AIME staff members found themselves right in the middle of the flood devastation caused by Hurricane Diane. The writer was in Wilkes-Barre, Pa. the weekend of August 20 and the freakish behavior of Diane was dramatically demonstrated in that area.

Wilkes-Barre was relatively untouched, but 14 miles to the north the city of Scranton was hard hit. The only inconvenience in Wilkes-Barre was the need to boil all drinking water for 20 min.

Unknown to us at the time Bob Shearman, AIME Metals Branch Secretary, was returning from Niagara Falls. He had attended a meeting of Niagara Frontier Section committee members planning the Reactive Metals Regional Conference for next March. He started from Niagara Falls at 11:00 pm under clear skies. The train trip usually takes 10 hr . . . but here in Bob's own words is the story:

"I was returning from Niagara Falls in a sleeping car on the Lehigh Valley Railroad when awakened by train officials announcing that there was a washout ahead and advising that buses would be on hand in 40 min to take us to Wilkes-Barre. The time was 7:45 am, and it was apparent that we had been standing still for a long time. The unfamiliar surroundings turned out to be Coxton Yard, 12 miles north of Wilkes-Barre. This was Friday morning, August 19. We soon learned that Hurricane Diane had passed through the night and had left her mark in no uncertain terms.

"The buses eventually arrived bearing passengers who had left New York 12 hr before and who had been taken from their train when further railroad travel through the Lehigh Valley had become impossible. Our train turned back to Buffalo and Toronto (to the consternation of our porter, who had expected his day off to be spent in New York!).

"Rail travel had become impossible because of washouts—some 13 of them. Buses took us to the Wilkes-Barre Station where after two more hours it was apparent that trains could not be run safely, and we headed for Lehighton by bus. The route was circuitous, with many evidences along the highway of heavy rainfall the night before. National Guardsmen prevented entry to Tamaqua, flooded by the Schuykill River.

"At Jim Thorpe, Pa. (formerly Mauch Chunk), we had our first glimpse of the raging Lehigh River —the immediate cause of most of the Lehigh Valley Railroad's difficulties. Across the river, railroad tracks were suspended in mid-air, with the bank washed out beneath. On our side of the river, the bus plowed through water 3 ft deep, with adjoining tracks of the Central Railroad of New Jersey awash.

"Upon arrival at Lehighton, possibilities of resuming our rail journey were ended. Water flooded the tracks and the station building had become an island. The railroad arranged for lunch to be served the three busloads of the faithful. It was now 2:00 pm or 15 hr since leaving Niagara Falls.

"Buses went on to Easton which not only had the Lehigh River to contend with but also the Delaware. The disastrous conditions at Easton have been well publicized. High water had isolated the Lehigh Valley Railroad Station, but eventually arrangements were made for a train to leave from the Central of New Jersey station. Time was now 8:00 pm—with two more hours of travel to New York. Originally scheduled arrival time in New York had been 9:00 am. We were just about 13 hr late!"

Bob arrived late but safe. We stopped in Easton on our way back. With the entire region under marshal law, armed troops complete with fixed bayonets patroled the streets. The main span of the old city suspension bridge had snapped under the strain of tons of debris—it looked bombed out. The area along the immediate shore was a shambles of furniture, homes, and cars. Cleaning up appeared to be a staggering task.

This shot was taken from the front of the bus as it plowed through 3 ft of water near Jim Thorpe, Pa. The Lehigh River which makes a right angle turn here is at the left.

After this brush with Diane, we can appreciate more deeply the experiences of AIME members in the flood disaster areas. Perhaps the hardest hit section of all was the Naugatuck Valley in Connecticut where approximately 40 pct of U. S. copper and brass production is centered.

A check with the Copper & Brass Research Assn. revealed that an almost unbelievably rapid recovery is being made among flood damaged companies. The biggest problem confronting the copper and brass mills is the cleaning and repairing of electrical installations. Tens of thousands of electric motors of all sizes must be cleaned, baked out, and checked. Intricate machinery and equipment, such as casting furnaces, rolling mills, extrusion presses, draw benches, annealing furnaces, and countless fabricating machines must be completely disassembled, cleaned, repaired, checked, and reassembled. The fine river silt penetrated everywhere and every piece of machinery must be inspected piece by piece.

When pictures of this destruction began to arrive here, most of us on the staff recognized plants and towns we had visited. It was in this sense a personal loss. We only hope that when we meet our friends from those areas again, they will be able to report that both homes and businesses are once more operating normally.

Ed Weiss

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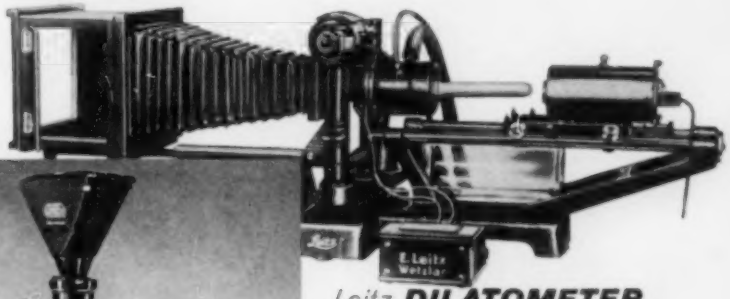

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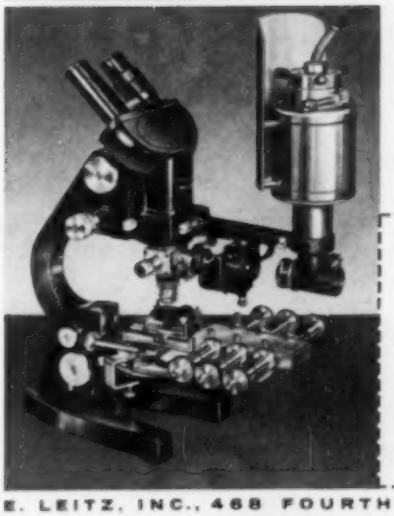

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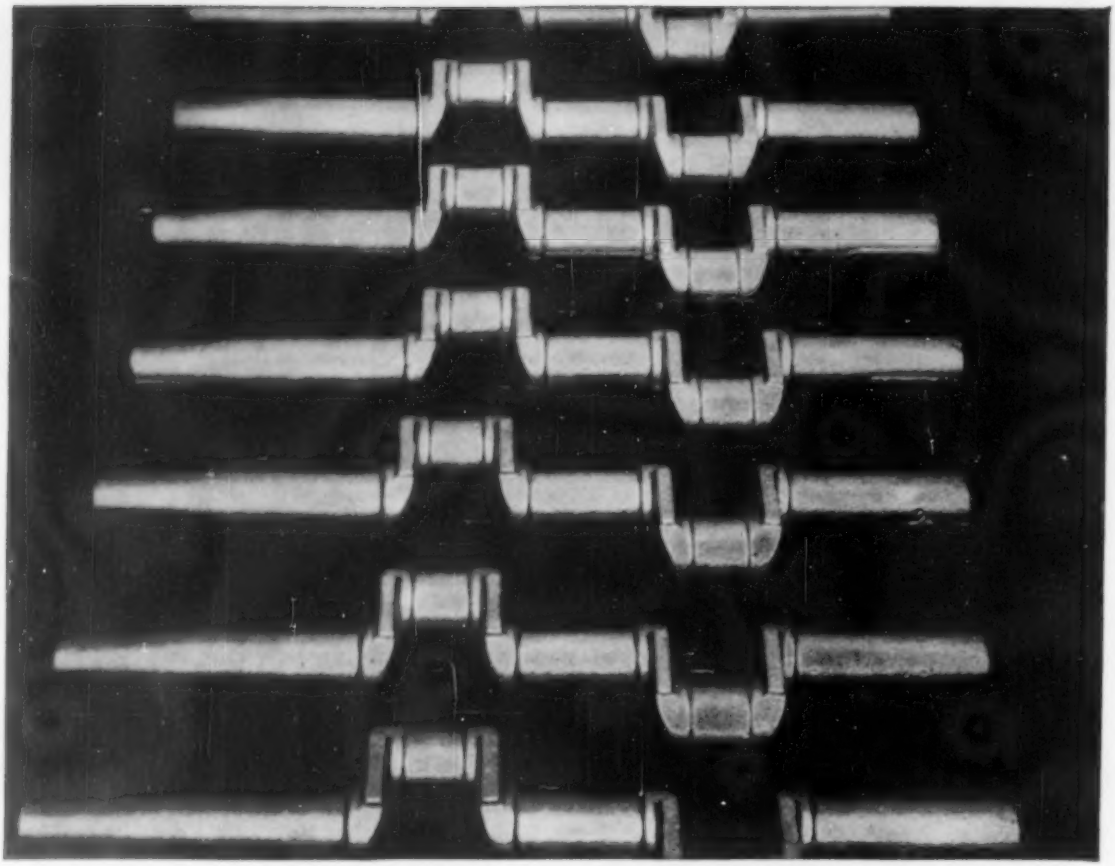

Ductile Cast Iron meets a variety of demands. These crankshafts utilize the 80-60-03 type, with the following typical range of properties: Tensile strength 90/110,000 psi, yield strength 60/75,000 psi, elongation 3/10%, hardness 200/270 BHN. Strong as-cast, tough and easily machinable. Also responds readily to flame or induction hardening. A carbidic, abrasion-resisting surface may be obtained by casting it against a chill.

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OTACO LIMITED, makers of farm machinery at Orillia, Ontario, formerly used crankshafts of forged mild alloy steel for 2-cylinder gasoline engines.

By switching to crankshafts cast in Ductile Iron, this manufacturer cut down some 11 machining and heat-treating operations to a total of only 5 machine operations.

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AEC plans to procure additional quantities of high purity zirconium and hafnium metal to meet the increasing requirements of its reactor development program. Tentative plans call for delivery of 2 million lb of zirconium metal over a five year period or 1.2 million lb over a three year period.

GE's Carboloy Dept., Detroit, will carry out the commercial development of the process for manufacturing diamonds. This GE unit was given the assignment on the basis of its metal-working experience associated with cemented carbides.

Ajax Engineering Corp., Trenton, Otto Junker Co., Germany, and Birlec Ltd., England, are pooling their technical resources and experience in developing and marketing the Line Frequency (60 Cycles) Coreless Induction Melting Furnace.

Allegheny Ludlum Steel Corp., prompted by the shortage of nickel, will continue further research on stainless steels made with manganese in place of nickel in order to assure future supplies of austentitic stainless steels.

Armour Research Foundation has developed a technique known as fiber metallurgy that involves the use of short metal fibers. According to A. G. Metcalfe, use of the technique can result in products having a wide range of porosities combined with relatively high strength and toughness.

Stanat Mfg. Co., New York, is building four 2-high combination rolling mills for AEC's Argonne National Laboratory, Lemont, Ill. Scheduled to begin operation in 1956, the mills will process plutonium-containing fuel elements essential to the development of reactor power plants.

Beryllium Corp., Reading, Pa., completed an expansion program that increased alloy capacity by 60 pct. According to L. F. Boland, vice president, demand for beryllium copper has necessitated the adoption of mass production and quality control equipment used in steel and aluminum mills.

West Germany produced 90,700 tons of virgin aluminum in the first eight months of this year as against 84,200 tons during the same period last year. Forecast for production in 1955 is 137,000 tons.

Reynolds Metals Co. has announced the use of a film of aluminum oxide 0.0002 in. thick to replace conventional insulating materials in electrical equipment. It has enabled the development of a new method of winding coils with aluminum foil or sheet insulated only with aluminum oxide.

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An informative book, entitled "Tungsten Steels", has been prepared by the Molybdenum Corporation. It deals with both scientific and practical considerations, and recommends procedures found to yield best results. A copy will be mailed on request.

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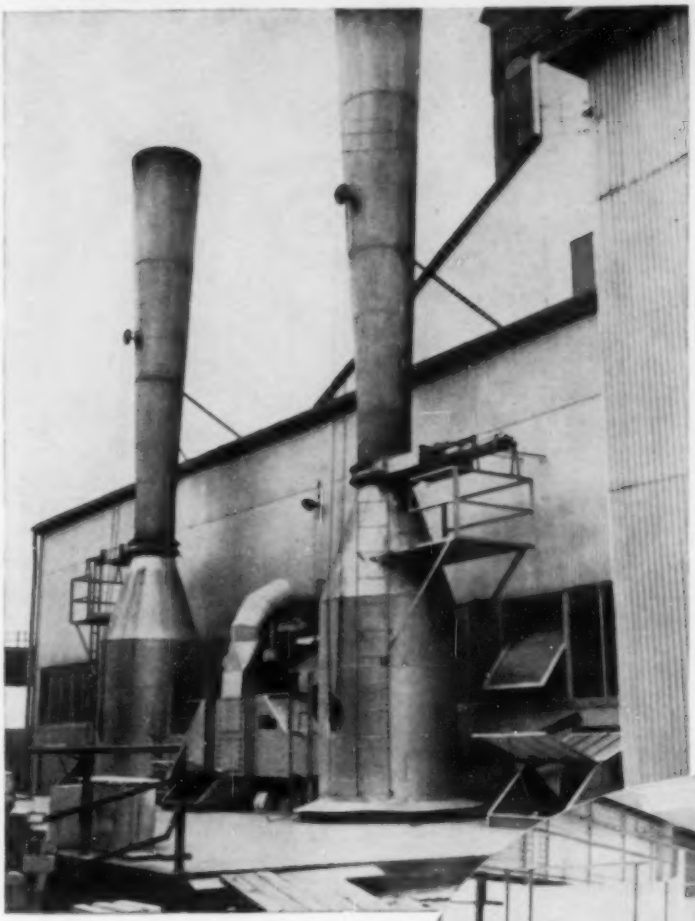
PRODUCTION UP- FUEL COST DOWN WITH MORGAN-ISLEY COMBUSTION CONTROLS



Morgan-Isley Ejector installation at Mississippi Glass Co., St. Louis, Mo., world's largest manufacturer of rolled glass. Inset shows one of Mississippi Glass products in use as office partitions.

In commenting on the Morgan-Isley installation at his company's St. Louis plant, Mr. Ellis Humphreys, Vice President of Mississippi Glass Co. said, "An ejector stack is far less temperamental than a natural draft stack, and, of course, is much less costly to build.

"One of the great advantages of the Morgan-Isley system comes from the secondary checkers. These give the incoming air a fine prewarming before it goes into the regular checkers. This not only results in better furnace operation, but seems to keep the regular checkers in much better condition. Incidentally, the secondary checkers seem to last indefinitely, and don't need to be cleaned during repairs. This system is good and greatly improves furnace operation."

CCC48

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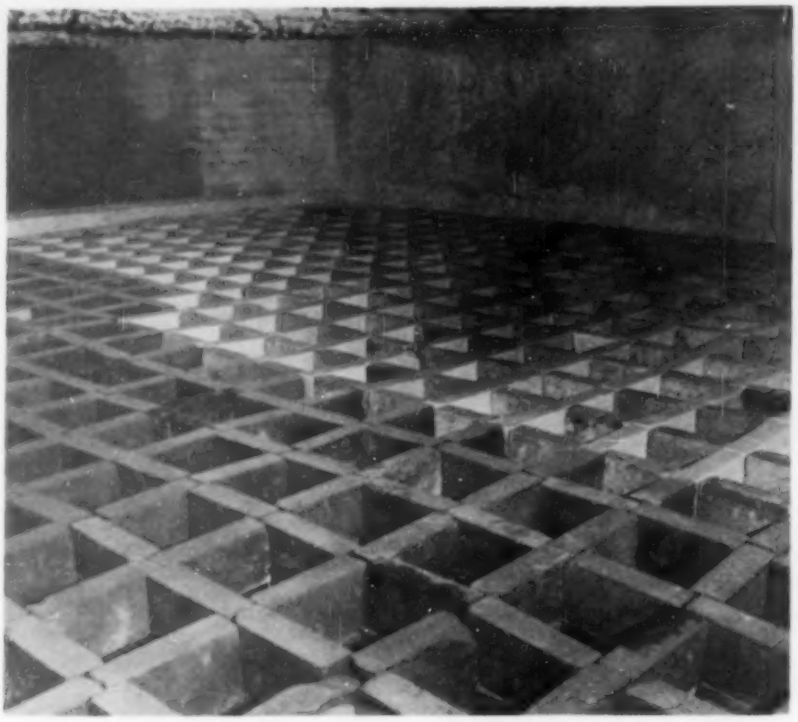


Fig. 1—Basic installation consisted of the top five courses of checkers of approximately 50 pct of the area of both ends.

Basic Checkers Proved Economical On Replacement Comparison With Clay

by C. C. Benton

EARLY in 1953 Algoma Steel Corp. realized the necessity of increasing firing rates for higher productivity per operating hour. No. 2 shop has four 150-ton furnaces. Nos. 21, 22, and 23 furnaces have a hearth area of 507 sq ft with fan capacity to furnish air for a maximum of 400 American gallons oil equivalent. No. 24 furnace has a 574 sq ft hearth area, and is equipped with an Isley Stack system with air and fuel capacity of 800 American gallons oil equivalent. However, this furnace was being fired at 400 American gallons maximum.

Because combustion air on furnaces 21, 22, and 23 could be increased to fire a maximum of 525 American gallons oil equivalent, the first part of the program was to increase to 525 gal per hr No. 24 furnace, the Isley unit.

C. C. BENTON is Superintendent, Open Hearths and Bessemer, Algoma Steel Corp., Sault Ste. Marie, Ontario, Canada. This paper was presented at the AIME National Open Hearth Committee meeting, Philadelphia, Apr. 18 to 20, 1955.

Checker replacement on all furnaces had been excessive, running normally 15 pct at approximately 300 heats and 40 pct at 600 heats.

Use of Basic Checkers

Because it was a physical impossibility to increase regenerator chamber area on Nos. 21, 22, and 23, a careful check was made of refractories for regenerative use that could be adapted for increased preheat capacity. A chrome-magnesia type of basic brick was chosen. To establish economies it was decided to make the first installation in the No. 24 Isley equipped furnace firing 525 American gallons oil equivalent.

Fig. 1 shows the first installation of basic checkers in March 1954. This furnace has single chamber regenerators. The basic installation consisted of the top five courses of checkers of approximately 50 pct of the area of both ends. Burned chrome-magnesite 9x4½x2½ in. was used and set up in a closed flue design having 7¾x7¾ in. flues. The remainder of

Table I. Comparison of Campaigns on No. 24 Furnace, Using Various Types of Checkers

Data	Checkers Used		
	All Clay	50 Pct Basic Capped	100 Pct Basic Capped
Fuel input, U.S. gal oil per hr	525	525	525
Heats	594	538	129
Avg heat size, tons	151	153	155
Avg time, charge to tap, min	604	530	510
Tons per hr, charge to tap	15.0	17.4	18.3
Btu's per ton	4,242	3,988	3,700
Pct hot metal	57.0	51.8	59.5

the chamber was 9x4½x2½ in. first quality stiff mud fire clay brick for the complete height. This material was also used under the basic courses.

As the campaign progressed it was apparent that furnace performance had improved. After two roofs the furnace, having tapped 293 heats, came down for a general rebuild including slag removal. At this time no replacement of basic checkers was required while 15 pct replacement was necessary on the clay side. There was some evidence of thermal shock in the top course of the basic section. Build up was very slight and readily removable with practically no dust accumulation below the top course. On the fire clay side dust accumulation was heavy and impossible to remove on top five courses.

After this observation it was decided to proceed with the experiment on one of the other 150 ton furnaces. Two basic brick manufacturers to recommend brick for this purpose, and two types of brick were recommended by each. Because the furnace chosen, No. 21, has double regenerators (air and gas) an installation of basic brick was made in the top five courses of both the larger or air chambers using all first quality stiff mud clay brick in the small chambers using same setting as on No. 24. One of the air chambers was given to one manufacturer, and the other to the second manufacturer. One half of one chamber was a high magnesia chrome brick and the other three sections were of three types of burned chrome-magnesia brick of various percentages.

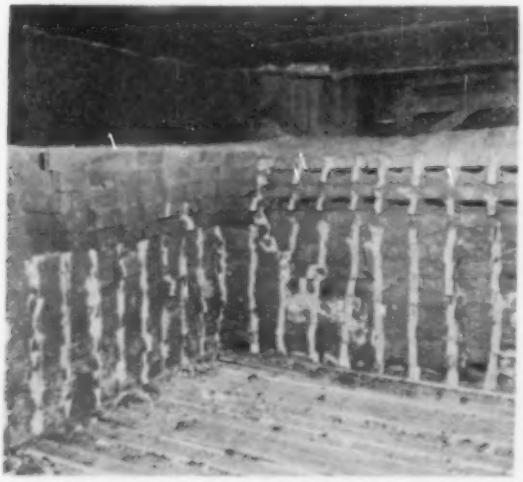


Fig. 2—A comparison of the condition of the clay and basic brick is shown after 538 heats.

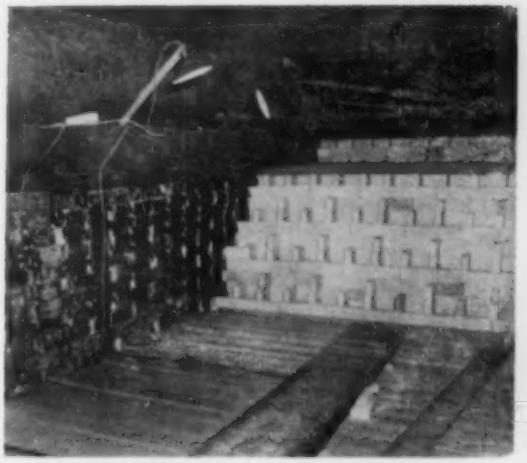


Fig. 3—The basic section is shown with the top course removed. Checkers were capped with a Hi Magnesia basic brick for further testing.

Operating Results Obtained

A decided improvement in operation was realized on No. 21 furnace. But because firing rates had also been increased, only the operating data on No. 24 furnace will be given. At 166 heats No. 21 furnace came down for a roof and front wall, and for inspection purposes the checkers were opened. It was found that the high magnesia type brick showed considerable cracking and disintegration on the top course, while the chrome-magnesia type were in perfect condition.

Because of the failure of the slag pocket walls it was necessary to take No. 24 furnace off at 245 heats. At this time there was a total of 538 heats on the checkers.

Fig. 2 gives a picture of the clay brick and the amount of removal required. It gives also a comparison of the condition of clay versus the basic.

In Fig. 3 the basic section after cleaning is shown with the top course removed. It would not have been necessary to remove all of the top course, but because economics had already been realized, comparing brick for brick with clay, it was decided to cap the balance of the checkers with a sixth type of basic brick (Hi Magnesia) for further testing.

Table I compares the same firing rate between two campaigns on clay checkers with two on the basic brick, and the continued performance of No. 24 furnace with basic capped checkers up to March 14, 1955. A lower fuel rate with a lower volume of hot metal was realized on the basic capped checkers.

Conclusions

Operating conditions as practiced at Algoma Steel Corp. have lead to the following conclusions in the use of basic capped checkers.

A—At 700 heats there is no dust adherence and slagging on the basic checkers below the top course.

B—Flues remain open without blowing for the entire campaign.

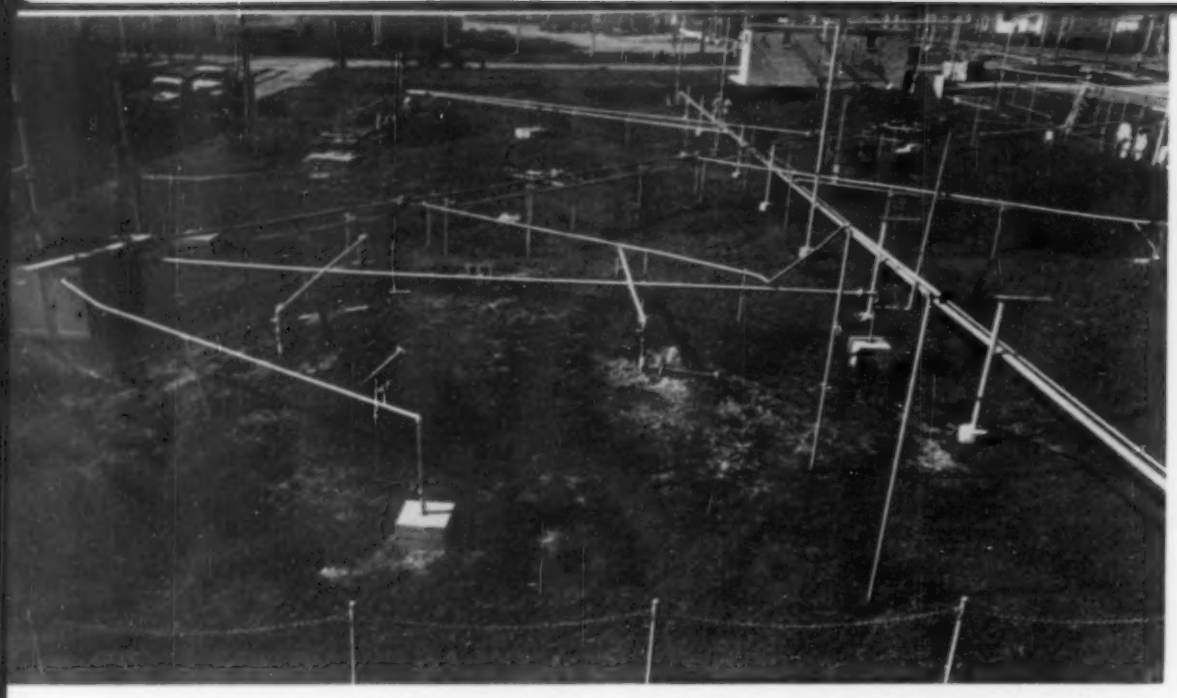
C—Clay checkers under basic are in good condition at 700 heats.

D—The trend is to higher production rates and lower fuel rates on basic checkers.

E—Basic checkers are economical on a straight replacement comparison with clay.

liquid and gaseous wastes, metallurgy and the ma-

original soil, or both, depending on the kind of con-



View of tank farm at Oak Ridge shows surface plumbing system associated with underground tanks holding radioactive liquid wastes.

Waste Disposal Is Vital To Atomic Power Development

by John M. Warde and Raymond M. Richardson

WHAT to do with atomic wastes is one of the major problems of the atomic age. Unlike other waste materials, these cannot be burned, evaporated, or filtered, and the transfer of radioactive material from one physical or chemical state to another is not a complete answer, because only time diminishes radioactivity.

Rate of decay of radioactive materials varies from a few seconds to several thousand years and cannot be altered economically by any known method. At present the Atomic Energy Commission and its contractors are coping with the situation adequately. Intensification of private development of atomic power could, however, pose problems in the nation's nuclear energy program.

One way to dispose of radioactive waste in liquid form is to release it into the ground. The problems of this method are basically geologic. Reliable information on the prevailing geologic and hydrologic features of the disposal area is fundamental in determining the effect of such releases in a given locality and in evaluating the potential hazards to water supply. Low level liquid wastes are sometimes discharged directly to the ground from operating disposal schemes. Radioactive contamination of the subsurface is also possible from accidental surface spills, seepage from liquid waste holding facilities, failure of underground liquid waste storage tanks, and from the underground leaching of buried solid wastes. Fall-outs from atomic weapon tests may find their way into the ground as a result of percolation.

In addition to the job of controlled release of radioactive wastes, there is the problem of protecting the environs of atomic energy sites in event of accidental discharge of radioactive materials.

Radioactive wastes arise from several sources, including chemical separation processes in reactor operations, reactor coolants, laboratory research on radioactive materials, concentration processes for

J. M. WARDE is Head, Ceramic Laboratory, Metallurgy Div., Oak Ridge National Laboratory, Oak Ridge, Tenn. R. M. RICHARDSON is Geologist, USGS, Memphis, Tenn. This article is abstracted from MINING ENGINEERING, May 1955, Vol. 7, pp. 458 to 461.

liquid and gaseous wastes, metallurgy and the machining of fissionable materials, and uranium ore processing.

Current methods for handling radioactive wastes are summarized in Table I. The principal problem in the safe handling and disposal of such wastes occurs in the chemical processing phase of nuclear reactor operations where unused uranium and plutonium are separated from fission products. Fluids used may be highly radioactive. Sufficient heat may be generated in the liquids by radioactive decay processes to cause boiling or evaporation. Characteristics of liquid process wastes are dependent upon the reactor system that produces them. Generally, they are uniform for a given system. In the case of a reactor where fissionable material is in the form of a soluble salt dissolved in water, waste would be comparatively free of solids and precipitants. On the other hand, process wastes from the acid dissolution of solid, aluminum canned uranium fuel elements will contain large quantities of dissolved aluminum nitrate and nitric acid.

Dissolved solids in reactor cooling water become radioactive and the liquid must be kept in storage to allow decay to safe levels before discharge. Liquid wastes produced in a laboratory are unpredictable both in composition and activity level. Laboratory waste liquids are monitored and if activity is high, stored in tanks. If low, they are discharged to the process-waste system and eventually flow into the ground or surface watercourses. Liquid wastes with dangerous levels of activity are placed in semipermanent storage.

Water used for cooling the reactors at the Hanford Atomic Products Operation, Richland, Wash., is discharged into the Columbia River after a period of storage. Low activity wastes are permitted to seep into the ground through *cribs*. The cribs consist of revetted pits. Radioactive elements adhere to soil particles as the waste infiltrates into the terrain. At Oak Ridge National Laboratory highly radioactive wastes are concentrated and stored in underground tanks. Low level wastes are first discharged into a settling basin, pass into White Oak Creek, to White Oak Lake, and finally into the Clinch River. Oak Ridge is experimenting with storing moderately radioactive wastes in large unlined waste pits or *lagoons*. This method, like that practiced at Hanford, depends on the ability of soil to remove activity as the liquid seeps through the zone of weathered rock and finds its way along bedrock to the White Oak Lake drainage system.

Oak Ridge handles its solid wastes by placing them in a trench and then covering with concrete, original soil, or both, depending on the kind of contamination. Brookhaven accomplishes this by packing wastes with concrete in sealed drums and dumping them at sea.

Gaseous and airborne wastes are normally diluted to safe levels upon release to the atmosphere. Careful meteorological checks are taken of prevailing conditions before releases are made. At Oak Ridge periodic readings taken of the air within a mile radius of exhaust stacks show activity to be about twice the average cosmic ray background—a safe condition and one representative of normal readings at other sites.

Geologic and Hydrologic Considerations

The ideal ground area for radioactive liquid waste release is one where the geologic conditions prevent migration of the materials or slow migration enough to allow radioactivity to decay to a safe level. Each site presents a distinct problem dependent on geologic and hydrologic characteristics. Before suitability of a site can be determined one must know the factors controlling the rate and direction of liquid flow into the subsurface. Practically all rocks are collections of discrete particles more or less perfectly cemented, rather than solid masses. Thus, it follows that nearly all rocks are pervious to fluids to some degree. Permeability of natural earth material varies greatly. Laboratory determinations show that gravel permeability may be 450,000 times that of a clayey silt. Much wider contrasts exist. Fluid contained in porous soils and rocks is not static, but is in slow, definite motion called laminar flow. Movement of fluid through a porous medium is caused by the difference of pressure head from point to point, in the case of ground water nearly always caused by gravity. Rate of movement depends chiefly upon the permeability of the rock, the hydraulic gradient, and temperature of the fluid. High temperature fluids usually flow at a rate greater than that of cooler fluids. Under normal conditions ground water flows at rates varying from a few feet per day to a few feet per year, although flows of several hundred feet per day may occur in cavernous limestones.

In unconfined or water table aquifers, fluids released above the water table infiltrate the soil and rocks below the surface, move down through the unsaturated region and enter the saturation zone. In the unsaturated region movement is generally downward. Some fluid may return to the surface by evaporation or transpiration. Slope or configuration of the water table usually conforms to the land surface. Fluid movement tends towards neighboring streams and lakes. While fluids introduced into un-

Table I—Handling of Radioactive Wastes in Atomic Energy Operations

Type	General Nature	Usual Treatment
Liquid	Metal waste containing fissionable metals; radiochemical waste containing high concentrations of radioisotopes; reactor cooling water; process water composed of water which is actually or potentially contaminated.	*Shorter half life wastes*—Monitor, store to permit radioactive decay to safe levels, dilute and release directly to surface water or to the ground or dispose of by incorporating in concrete and burial at sea. *Longer half life wastes*—Concentrate by evaporation, filtration, precipitation, crystallization, or ion exchange and place in semipermanent storage in underground tanks.
Gaseous or Airborne	Radioactive rare gases, chemically reactive gases, and radioactive solid particles.	Filtration by mechanical separators and electrostatic precipitators. Where necessary filtration is preceded by scrubbing. Filtered gases released to atmosphere through tall stacks under meteorological control.
Solid	Contaminated clothing, process equipment, building materials, laboratory apparatus and glassware, biological specimens, air filters and solidified liquid wastes.	Except for some closed cycle incineration of combustible wastes, bulk of solids are disposed of by burial in the ground or by incorporation in concrete and burial at sea.

confined aquifers may return to the surface near where they were introduced, in regions where rocks of the unsaturated zone are comparatively dense or subsurface frozen, fluid percolation is retarded. Other regions may consist of more permeable material and downward movement is more rapid. Impervious strata above the main water table could support perched saturated zones.

Ground water is confined under hydrostatic pressure where artesian conditions exist. Water bearing strata are covered by less pervious rock penetrated only with difficulty by downward percolating fluids. Recharge to such acquifers occurs chiefly in or near their updip outcrops, from which the fluids move to discharge points. Direction and character of flow in confined aquifers is relatively independent of detailed surface topography, although ordinarily movement is from the higher to lower topographic levels underlain by the aquifer.

One element to be considered in the underground migration of radioactive wastes is the disruptive effect on the normal hydraulic gradient caused by withdrawal of water from wells. When a well is pumped water levels are drawn down in the vicinity and a cone of depression is formed. Movement from all directions toward the well is induced by the hydraulic gradient. As pumping continues the cone expands and movement is induced from increasingly distant points. Rate of withdrawal, hydrologic properties, aquifer dimension, location of areas of recharge and discharge determine the size, shape, and rate of growth of the cone.

Difference in density between liquid waste and ground water or other natural fluids influences the rate and direction of waste movement. Fluids with greater density than the natural fluids in the formation will move downward through the ground water as a mass. Wastes lighter than the natural fluids will be buoyed up by them.

An important role is played by dilution in safe disposal of liquid wastes into surface watercourses, but apparently is a less important factor where ground water is concerned. Knowledge of the mechanics of flow of fluid wastes through permeable material saturated with ground water is at present imperfect. However, under conditions of laminar flow contaminants move essentially as one stream with relatively little dilution.

How much radioactivity from a migrating waste solution is fixed in the enclosing rocks and the permanence of the fixation when the rock is alternately wetted and dried by filtration or by fluctuation of the water table is important in control of underground activity movement. Sorption properties of clays are significant where fluid wastes are released to surface waters, since the turbidity in a water body may alter distribution of the activity and the sediment on settling may increase activity on the bottom.

Chemical Interactions

The possibility of chemical interaction between liquid waste and ground water, other natural fluids, or the enclosing rock is a major factor in evaluation of migration activity. Permeability reduction and eventual plugging of rock pores can be caused by presence of suspended solids, colloid swelling, aggregate slaking, bacterial growth, or precipitants. Liquids containing calcium salts could precipitate calcium carbonate and calcium sulphate when coming in contact with dissolved carbonates and sulphates. Acid wastes traversing carbonate rocks may develop or enlarge solution channels in such rocks.

Liquid wastes arising from processing aluminum canned uranium slugs, assuming it contains aluminum nitrate and nitric acid, coming in contact with calcareous rocks would form a gel which could seal surrounding rock. Nitrate in high concentrations constitutes a potential health danger. Many public health authorities hold the opinion that drinking water containing more than 10 to 20 parts per million of nitrate nitrogen may be responsible for methemoglobinemia. Infants with this disease are called blue babies.

Decaying radioactive liquids may generate considerable heat. Heat removal rate by surrounding rock is dependent on the thermal conductivity of the rock and its contained water.

Site Investigations

In seeking a site for radioactive liquid disposal all sources of information should be integrated—geologic, lithologic, stratigraphic, and structural features, private, state, and federal topographic, geologic, and hydrologic maps and reports—in particular, information on ground water occurrence.

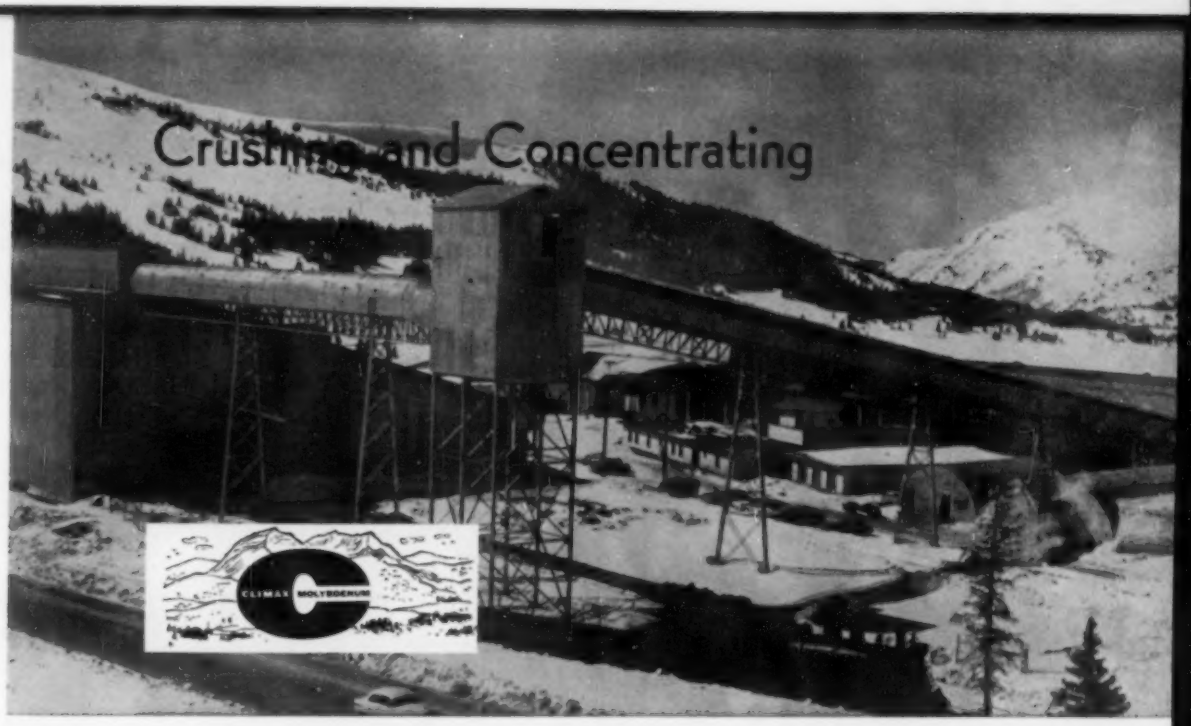
Test drilling should be carried out to determine ground water conditions and define the lithologic and structural features of the subsurface. Exploratory drilling should reveal perched water, confining beds, and other flow barriers, faults, fissures, and other openings permitting the flow of liquid contaminants. Electric logging is a useful adjunct to drill cores or cuttings. The program would provide samples for petrographic examinations, isotope-retention studies, radioactivity determinations, chemical interaction tests, and measurements of hydrologic properties. Holes can serve as observation wells for water-level measurements and monitoring of radioactivity by radio logging. Under the heading of hydrologic investigations come permeability tests, water-surface mapping, and determination of the rate and direction of ground water movement. Liquid wastes released to surface water should be hydrologically studied for the rate of dilution under various conditions. Velocity measurements in the case of flowing streams and sampling to determine dispersal pattern of the activity in the water body are necessary in the latter case.

Research and Development

Geologic aspects of the disposal of radioactive wastes are part of the program supported by the AEC. Detailed geologic and hydrologic studies are underway in cooperation with the U. S. Geological Survey at all installations where radioactive material handling is a problem. More work is needed. Many branches of science are already involved in research on the mechanisms of retention of radioactivity by certain minerals and development of methods for permanent fixation of radioactivity in the ground. Reports from Brookhaven on the fixation of radioactive materials on clay by sorption processes and subsequent heat treatment to make the action irreversible have been encouraging. Also under consideration is the possible disposal of radioactive wastes in abandoned mines, deep wells, and salt domes.

Acknowledgments

Thanks are due to Conrad Straub, U. S. Public Health Service, members of the health physics div., Oak Ridge National Laboratory, as well as W. K. Eister, chemical technology div., ORNL, and T. N. McVay and J. R. Johnson of the ceramic laboratory, ORNL, for guidance and review of the manuscript.



MILLING operations at Climax started in 1918 with a 200-ton flotation plant. At that time the crusher was located at the mine and ore was delivered to the mill on a tramway. The plant closed down in March 1919 and remained closed until 1924, by which time a modest demand for molybdenum had been developed. From 1924 to 1932 the mill capacity was increased to 1200 tons per day, and mill sections added in 1932 increased capacity to 4000 tons per day. In 1936 and 1937 six additional mill sections were built, bringing the designed tonnage to 10,000 tons per day. One more section was added in 1940. This together with improvements in grinding technique increased tonnage to 18,000 tons per day. The most recent expansion program includes six more mill sections and the Storke level crusher. Present capacity is 28,000 tons per day.

Crushing Done in Three Plants

Crushing of Climax mine-run ore is carried out in three stages. No. 1 and 2 crushers are used for ore from the Phillipson level and the No. 3 crusher treats ore from the Storke level. The flow of materials is the same in all three, but the third stage of crushing for the Storke level crusher is done in No. 2 crusher.

Equipment in No. 1 and 2 crushers is identical, but the capacity of No. 1 crusher is 5000 tons per day while the capacity of No. 2 crusher is 14,000 tons per day.

Phillipson ore is dumped into the crusher bins from 10-ton Granby cars and fed to 48x60-in. Buchanan jaw crushers through chutes and Ross feeders. The jaw crushers have a discharge opening of approximately 9 in. and a capacity of 500 tons per hr each. The product from these jaws is carried by pan and belt conveyors to a 5x9-ft vibrating grizzly with 1½-in. openings. The oversize is fed to 7-ft standard Symons cones and the undersize joins the crushed material from the standard. The standard cone crushers are set at 1⅛ in. on the closed side and the product, all −3 in., is fed to storage bins. Operating in closed circuit with vibrating screens, 7-ft shorthead Symons cones prepare the final mill feed, averaging 6 pct on ⅜-in. mesh.

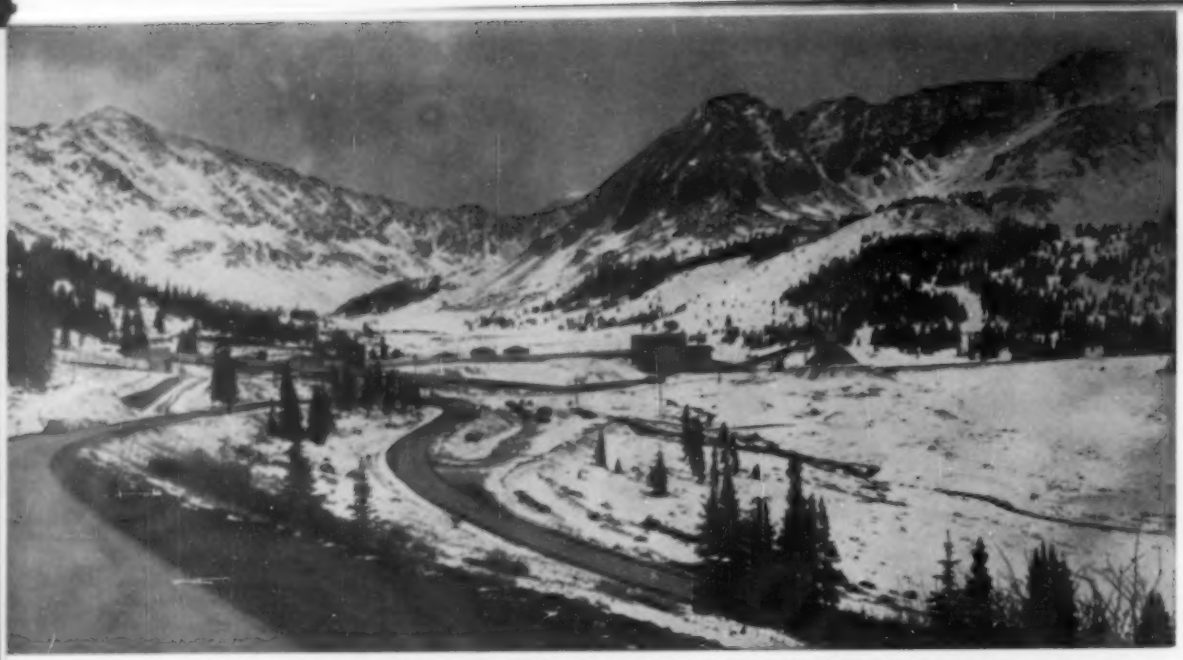
The Storke level crusher is designed to crush the entire mine production after the ore from the Phillipson level is depleted. The primary crusher at the Storke level is a 60-in. Nordberg gyratory. The main shaft complete with mantles weighs 90 tons and the total weight of the crushing unit is 500 tons. The crusher is driven by a 500-hp motor with a V-flat pulley arrangement. Countershaft speed is 240 rpm, driving the eccentric at 80 rpm. To guard

The Climax Expansion

The story of the Climax Molybdenum Co. expansion as presented here is a condensed version of the complete story featured in the August 1955 issue of MINING ENGINEERING. Emphasis has been placed on the articles that would be of special interest to the readers of JOURNAL OF METALS.

The authors of the original articles were: "Crushing and Concentrating" by F. Windolph and M. W. Dessau, Assistant Mill Superintendent and Mill Superintendent; "Climax Expands Ore Treatment Research" by R. E. Cuthbertson and F. J. Hoff, Research Engineer and Research Metallurgist; "Planning and New Construction" by M. Gelwix and J. W. Goth, Planning Engineer and Metallurgist; "Widespread Use of Synchronous Motors Gives High Power Factor" by U. F. Toucher, Chief Electrical Engineer; "Training Program" by H. R. Moody, Training Manager; and "Climax Uranium Co." by M. L. Kay, Vice President and General Manager.

Expanded versions of the following articles which appeared in MINING ENGINEERING will be published at a later date: "Research and Sales—Key to Climax Growth" by R. E. Warriner, "Detroit Research Laboratory" by A. J. Herzig, "Langeloth Conversion Plant" by E. S. Wheeler, and "Molybdenum Works for the Mining Industry" by T. E. Norman.



New Storke level crusher located 1500 ft from the Storke level adit, which is to the left of the picture on the same elevation.

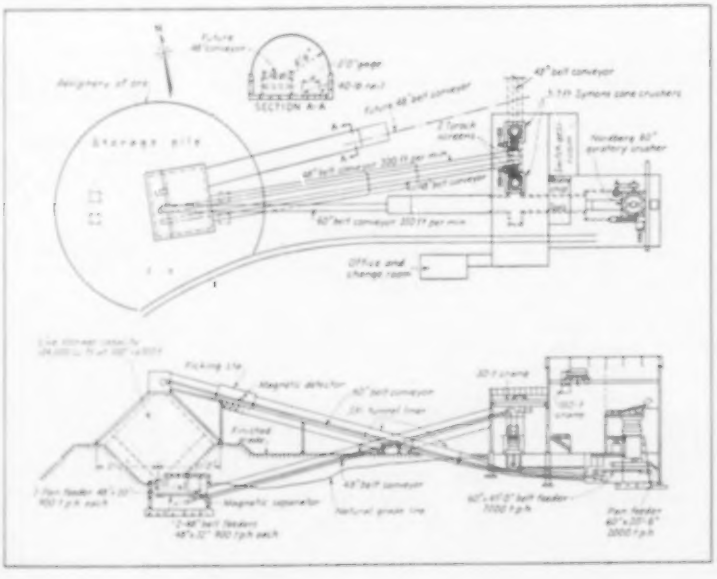
against oil failures the crusher is amply protected by flowmeters, high-low temperature gages, and pressure gages. These protective devices are interlocked with the drive motor so that any oil failure will stop the crusher. The machine cannot be started until the cause of failure is corrected. This crusher was started in February 1953 and to date neither the mantles nor the concaves have been worn out. However, the practice of welding manganese steel bars to the lower mantle appears to be profitable. This crusher has a capacity of more than 2000 tons per hr and delays due to large rocks are negligible.

This compares to about 20 pct lost time because of large rocks and hangups in crushers and bins.

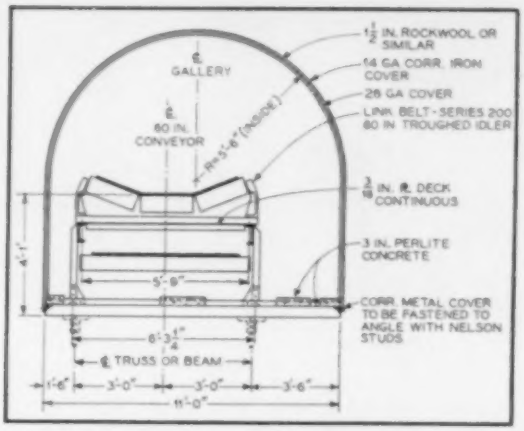
Ore from the primary crusher is stored in a coarse-ore bin adjacent to the crusher building. From this bin the ore is fed on a 5x9-ft vibrating screen, with 2-in. openings. The oversize product from the screen is crushed in a 7-ft Symons standard cone and added to the screen undersize.

The product from Storke crusher is transported nearly 4000 ft and elevated more than 300 ft to the fine ore bins near No. 1 and 2 crushers. This conveyor system is divided into belts of nearly equal length operating 475 fpm. Designed capacity is 1500 tons per hr. The ore from the fine-ore bin is delivered to the storage bins in No. 2 crusher for the final stage. The crushed ore from all three crushers goes to 14 cylindrical ore bins, 35 ft high and 35 ft diam, having a capacity of 600 live tons of ore.

Climax ore is an altered and highly silicified granite, and the most important mineral, molybdenite, is finely disseminated and found in thin veins



Many features are incorporated in the new Storke crusher, including bins constructed with earth fills and outside conveyor belts enclosed with semicircular corrugated-tunnel support. This crusher will handle all mine production when mining on the Phillipson is complete.



Section through an exposed conveyor gallery, showing insulation and lightweight construction. The curved corrugated covering is used for highway culvert support. Electric strip-heaters are installed in the gallery to keep temperatures above freezing in the winter.

and stringers associated with quartz. Other minerals present in the ore are wolframite, cassiterite, topaz, monazite, pyrite, and calcopyrite. Molybdenite is not difficult to float; however, the mineral in the Climax ore is very finely disseminated and good recovery at coarse grinds is one of the major problems. At present, the rougher concentrate is being floated at 43 pct +100 mesh. It is believed that this is one of the coarsest grinds of any large mill is existence. The ratio of concentration of rougher concentrate to total feed is nearly 25:1 and the rougher concentrate only requires fine grinding for further processing. This system confines the expensive fine grinding to a small amount of the total feed.

Rougher Circuit

The rougher circuit consists of 14 units in parallel. Each unit is made up of a bin, ball mill, classifier, three 13-cell flotation machines, and auxiliary equipment such as feeder belts, pumps, and blowers.

There are eight 9x8-ft grate-type Marcy ball mills driven by 450-hp motors and six 9x9-ft grate-type Marcy ball mills driven by 600-hp motors. The use of shiplap liners is standard practice and 3-in. forged steel balls are charged daily. Consumption of balls is 1.30 lb per ton of ore treated.

The classifiers are 78-in. Akins duplex high weir, with a double spiral. The first of these classifiers had 12-in. shafts, but recently completed installations have 16-in. shafts and it is felt that this increase in diameter will reduce shaft breakage. The circulating load is about three to one.

Although several types of flotation machines have been tried, the standard equipment is the Weinig subaerated hog-trough type. The power requirement for this machine is less than 1.0 kw hr per ton and air at 1¾ psi is used at the rate of 35 cfm per spindle. Normally the flotation circuit consists of three 13-cell flotation machines in series. The froth from the first machine is the rougher concentrate and the froth from the second and third machines is returned to the first. Table I gives the reagents used in this circuit.

Cleaner Circuit

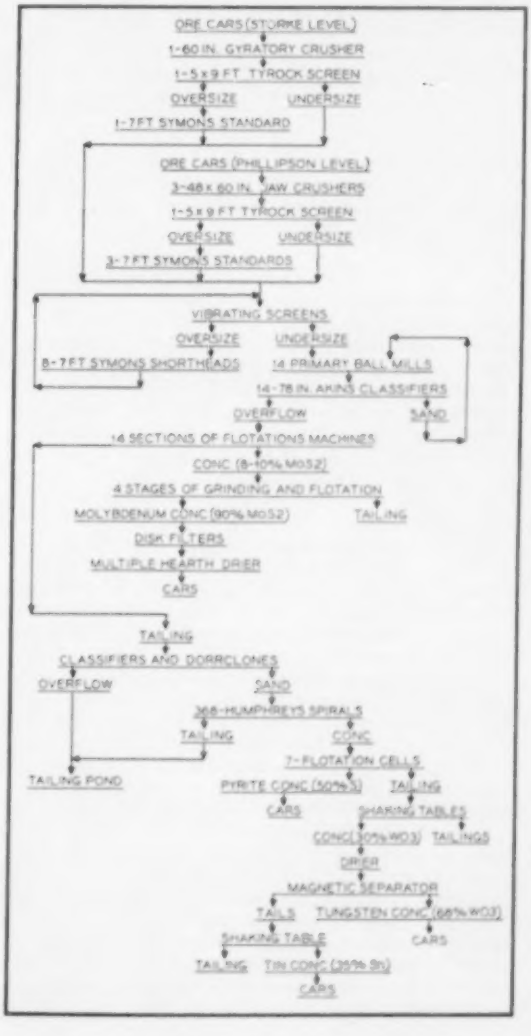
The rougher concentrate, containing 8 to 10 pct MoS_2 is thickened and then cleaned in four stages

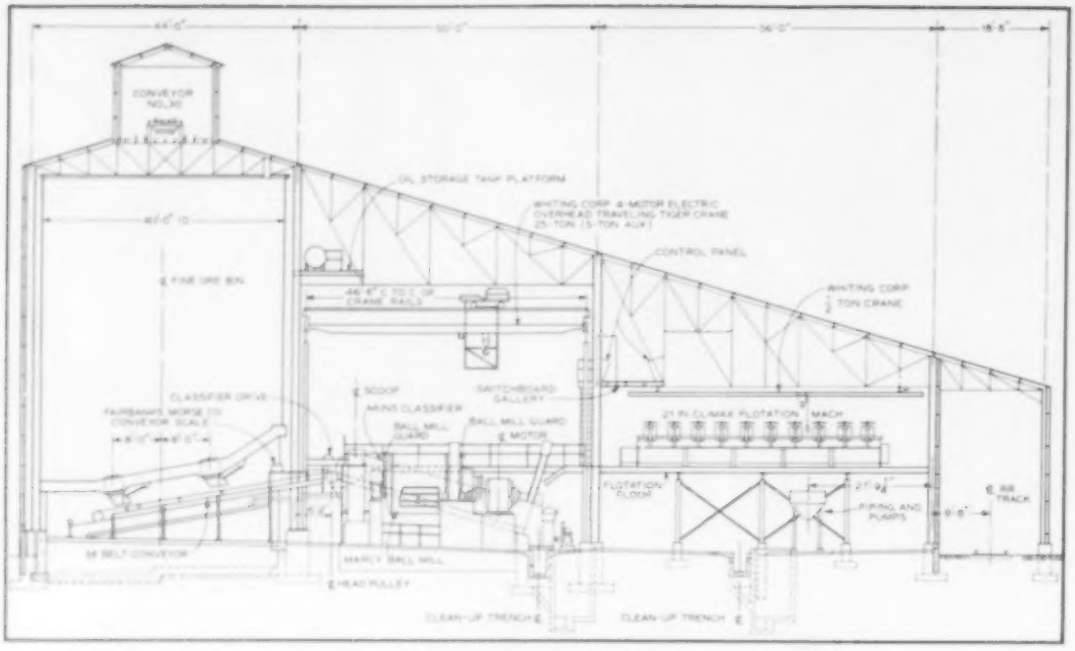
Table I. Reagents Used for Flotation Circuit

Reagent	Lb Per Ton
Hydrocarbon	1.00
Syntex	0.02
Pine oil	0.05

of flotation with grinding and classification between each stage. The first stage grinding in the cleaner circuit is done with two 5x20-ft mills charged with ¾-in. forged steel balls.

Second stage grinding is done with two 8x20-ft mills charged with 2 to 3-in. Texas pebbles. The final two stages of grinding use one 8x20-ft mill each, and the same pebble size as the second stage. Very fine grinding with flint pebbles seems to have no ill effect on subsequent flotation, but the same amount of work with steel balls will lower the recovery. The only reagents used in this circuit are small amounts of pine oil to promote molybdenite recovery, and soda ash with cyanide to depress the pyrite and calcopyrite. This cleaning circuit is in





Cross-section of No. 3 Mill

effect a complete mill in itself from which a tailing and the final concentrate is produced. Grade of concentrate is easily maintained and recovery seldom drops below 98.5 pct.

Filtering and Drying

The concentrate from the cleaning plant is filtered, dried, and finally loaded in hopper cars or barrels, according to the demands of the customer. The recently completed filtering and drying plant consists of two 6-ft Eimco disk filters. The filtered concentrate feeds a 20-ft four-hearth drier. The system replaced four drum-type filters and four oil-fired Lowden driers. The new scheme not only improved results but also reduced costs.

Byproducts

Finely ground tailings from the cleaning plant go to waste, and the coarse tailing from the rougher circuit is the feed to the byproducts plant. The most important mineral recovered from the rougher tailings is wolframite, but some cassiterite and pyrite are also obtained.

The presence of extremely small amounts of wolframite in the ore had been known for many years, but no economical method for its extraction was known until after the development of the Humphrey spiral. Test work was started with spirals in 1946, a pilot plant was built in 1947, and finally a 7000-ton unit was placed in operation in 1948.

This section of the mill has increased in size along with the rest of the plant, although the byproducts plant is overloaded at the present time. The flow of materials now used has evolved from the original 7000-ton plant and consists of spiraling classified products, removing pyrite by flotation, tabling the nonfloat from the pyrite flotation, removing monazite from the table concentrate by flotation, and upgrading WO_3 concentrate with magnets.

The Humphrey spiral is the key to the successful operation of the byproducts plant. The feed to the spirals contains about 0.025 pct WO_3 and the spiral concentrate contains 1.0 pct WO_3 after cleaning. Tonnage to each spiral is 40 tons per day. Recovery of the spirals is nearly equal to table recovery, but tonnage is double and requirements for floor space are about 40 to 1 in favor of the spirals.

The spiral concentrate contains about 50 pct pyrite which is removed by flotation in an acid circuit with xanthate, pine oil, and Dowfroth. A clean pyrite concentrate containing not less than 50 pct sulphur is produced and sold for acid manufacture.

The nonfloat from the pyrite flotation is tabled and the concentrate, containing about 25 pct WO_3, is fed to the monazite removal section where monazite is floated in a high acid circuit using amine to promote the monazite and starch to depress the wolframite. The nonfloat from this circuit, containing about 40 pct WO_3, is upgraded on magnets to 65 pct WO_3. The nonmagnetic material contains the tin, which can be upgraded by tabling or screening.

The tailings pond, located about three miles north of the mill, serves two purposes—tailing storage and water reclamation. Tailing is carried from the mill through 24-in. wood stave, machine-banded pipe. The crest of the present pond is 300 ft lower than the mill. The pipeline is laid in nearly horizontal sections, each succeeding section being at a lower level. The horizontal sections are connected by vertical drops open at the top. The length of these sections and the height of the vertical drops conform to the topography and are arranged so that each 1000 ft of horizontal pipe has at least 15 ft of vertical drop. These penstocks impose the necessary head to maintain the minimum velocity required to carry the tailings. During the summer months the tailings are distributed along the crest of the dam through 4-in. leads. The height of the berm built

ABOVE—Looking northwest across the Climax tailing pond. In foreground is 24-in. tailing line showing standard drop for maintaining head in the line. RIGHT—Six-pole magnetic separator producing tungsten concentrate in byproducts mill.

in this manner is raised still higher with bulldozers. During the winter the tailings are distributed through four or five 20-in. leads in front of the berm built during the summer. The water impounded in the dam goes by gravity or is pumped to Robinson Lake. This lake, located 3½ miles from the mill and 600 ft lower, makes up the mill water supply. The lake is filled in the spring by a system of canals that gather the spring runoff from the Ten-Mile watershed. During the winter, overflow from the tailings pond goes by gravity to Robinson Lake, but the seepage from the pond must be pumped. Consumption of water in the mill is about 1000 acre-ft per month, and since the capacity of Robinson Lake is only 2000 acre-ft, conservation of water is extremely important.

Climax Expands Ore Treatment Research

AS a part of its western operations, Climax, since 1926, has conducted a program of ore testing at the Golden laboratory of the Colorado School of Mines Research Foundation, which is staffed with a technical group directly employed by the company. Exclusive use of certain laboratories together with the joint use of all common plant facilities is established by contract with the Colorado School of Mines Research Foundation. The Foundation is a nonprofit corporation legally distinct from the Colorado School of Mines but closely associated with the school. The experimental plant, center of activities of the Research Foundation, is school property but is primarily used for research work sponsored by the mining and metallurgical industries.

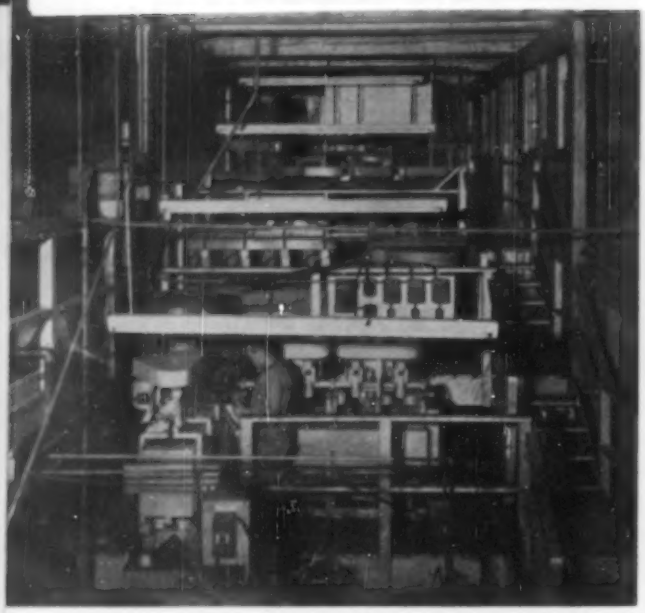
Recent expansion by Climax of research activities in the ore dressing field is the result of definite company policies. In the first place constant effort is being made to achieve maximum recovery of molybdenite and to produce concentrate of highest possible purity. Second, the management-inspired investigation of the recovery of byproduct minerals, which led to the installation of a plant in 1948 for recovery of tungsten concentrate, is still a very active undertaking. Third, new and varied demand for ore treatment investigation has been injected into the research program by the exploration dept.

Expansion of the volume of research work has been accommodated by increasing the Golden laboratory staff and establishing a new testing laboratory at the Climax mill, which operates as a division of the mill dept. A number of special projects have been placed with research institutions and universities to supplement direct company effort. These sponsored investigations in ore processing are carried out either under the direction of the company laboratories or under the general supervision of a company official who coordinates such work with the activities of testing staffs at Climax and Golden.

Mill Laboratory Attacks Operating Problems

The metallurgical research program was expanded in August 1954 by the installation of laboratory facilities at Climax. Being located at the plant, the laboratory personnel have access to the various plant pulps. This makes it possible to carry on investigation for the possible improvement of existing processes involving the recovery of molybdenite, tungsten, pyrite, and tin.

Initial molybdenite flotation testing confirmed



ABOVE—Pilot plant in Golden laboratory for testing magnetic separation used in Climax byproducts plant. LEFT—Molybdenum flotation test mill to check recovery and methods for possible application to the Climax flowsheet.

previous work by the Golden laboratory that there was a considerable spread between the recoveries obtained in the laboratory batch flotation cell and the plant flotation cells. Laboratory tests resulted in higher recoveries. It was possible to cut this recovery loss almost in half by changing the plant flowsheet to conform with the laboratory procedure. This new procedure decreased the grade of rougher concentrate about 2 pct as a result of a flowsheet change that sent all froth to rougher concentrate instead of recirculating the middling fraction back to the head of the circuit. Froth removal must be controlled at a slow rate to hold rougher concentrate tonnage and grade within limits of the regrind-cleaner section of the mill.

The remaining difference in recovery is attributed to the ability of the laboratory flotation cell to float a coarser middling particle than can be floated in the plant machines. As this difference in coarse particle flotation is believed due to mechanical aspects of the flotation cells an investigation is being initiated in conjunction with the operating staff in an effort to improve the performance of the plant flotation cells.

Climax management has adopted an aggressive policy of placement of research projects with institutions outside the company. From an ore treatment standpoint, investigations have been undertaken or are in progress covering a wide range of subjects.

Expert Analytical Work Essential to Research

By way of illustrating the nature of the analytical problems posed by ore treatment investigations a few figures are given from actual experience. Climax ore averages about 0.5 pct MoS_2 or 0.3 pct sulphide Mo. Consequently, a 1.0 pct improvement in recovery is measured by a lowering of mill tailing assay for MoS_2 content amounting to 0.003 pct. Obviously it is necessary to obtain reproducibility of assay results within that limit if test work is to have any significance.

In the case of tungsten a difference in tailing assay of 0.001 pct WO_3 measures a difference in recovery of approximately 4.0 pct. Accuracy of about this order has been achieved on low assay samples by the development of a special colorometric method for tungsten, which makes it possible to evaluate test work within the limits indicated.

Exploration activities also create some interesting analytical problems since ores of many types and from many localities flow into the research laboratory. So long as the engineers and geologists in exploration remain active there is little chance that the research laboratory will curtail its functions.

Planning and New Construction

THE planning dept. functions as a service department, its existence justified by the aid it can give operating departments throughout the plant. This aid comes through testing and selecting equipment and materials; plans and estimates for construction work; determining the merits of suggestions from various sources, which may range from management to the laborer; and working out methods of operation which are superior to past methods from a standpoint of time, economy, or safety. All of these things are of importance to the operation and help the production man who does not have sufficient time to investigate the necessary details.

At present this department is composed of a planning engineer, a metallurgist, a mining engineer, three test engineers, and a secretary. Problems

handed to these men cover a wide range of activity and may come from any department head or from management. The group is working on installation of a mill crane; converting from sack to bulk cement in the mine; building a jumbo and steel forms to replace wooden forms for loading cutouts; converting the main warehouse storage from wooden to all-steel construction; providing a sleeping room in the hotel annex with a 30 pct oxygen atmosphere; and working out a more economical means of supporting slusher drifts. These are a few of the projects now on tap.

Attacking Crushing and Milling Problems

Planning assists both the milling and crushing departments in solving prevalent metal wear problems. This phase of activity has a two-fold function: it assists the operating departments as well as aiding the sales dept. staff in testing new materials as they are recommended.

Climax ore presents a severe wear problem in grinding. Annual records of wear life of various materials have been kept by the operating departments and these records are being expanded and the data used in conjunction with various new materials that are being tested. Specific wear problems are hard to duplicate in a laboratory, and materials are therefore being tested in actual service under standard operating conditions. The function of the planning dept. is to correlate and supervise the testing program and to supply the results of the program to the interested parties.

Wear is a problem in all phases of crushing and grinding. Testing programs are either planned or have been completed on jaw crusher plates, chute liner materials, screen rods, cone crusher lines, ball mill liners, grinding balls, and classifier wear shoes.

Three of the new 18-unit apartment houses for resident employes. Overhead is a portion of the 4000-ft conveyor belt from the Storke crusher to the fine ore bin. This is typical of the construction jobs completed in the last five years.

Every man in the department understands fully that cooperation with the other Climax departments is of utmost importance, and that his principal duty is to aid the operating departments in every way possible. An engineer in this department soon finds that his work does not encompass routine and standard jobs.

It is difficult to measure the value of any service department. Many of the Climax projects are for long-range planning, but the target is to show, each year, a saving which is several times the departmental operating costs.

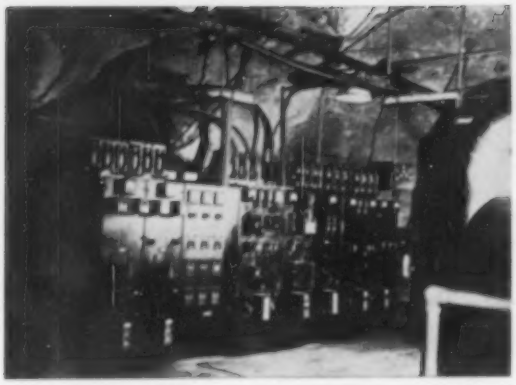
Widespread Use Of Synchronous Motors Gives High Power Factor

ELECTRIC power is the main source of energy for mining, crushing, milling, conveying, and processing ore at Climax. Power purchased from the Public Service Co. of Colorado is delivered over two transmission lines at 115,000 v to the Climax substation, where six 3333-kva single-phase transformers step it down to 13,800 v. From this main substation power is distributed through two main circuits to strategically located substations. Intermediate transformers have 440-v secondaries going to the switch and control centers at various points of utilization.

Underground Power

A 4/0 3-conductor steel wire armored cable rated at 23,000 v brings power to the underground operations. The cable is installed in the Storke level ventilation raise and terminates at a 3-pole air-break transfer switch. One side of the transfer switch is connected through an oil circuit-breaker to the Storke level substation. The other side of the transfer switch is connected through an oil circuit-breaker to the Phillipson level system, with a 4/0 3-conductor steel armored cable installed in the borehole from the Storke level to the Phillipson level switching station. Power is distributed from the switching station to three underground substations located as near the load centers as physical conditions permit.

Underground power at 440 v is distributed by 350 M cir mils cable from manually operated circuit-



Underground switching station that supplies power to electrical equipment is shown on the Phillipson level.

breakers. Total connected load underground is 27,500 hp with a calculated load factor from 30 to 40 pct. All circuits are adequately protected through coordinated tripping schemes.

Direct current power is provided at 275 v for the haulage system. The motor generator units all have flat characteristics and are paralleled by the use of automatic reclosing tie breakers, and the dc system is adequately protected by sectionalizing breakers. Main haulage trolley wire is 6/0 figure eight, supplemented by 500 M cir mils feeders, while laterals utilize 4/0 trolley wire and the return circuit consists of bonded trackage with a supplementary 500 M cir mils feeder. There are 44 traction units for the operation, ranging from 1½-ton trammers to 20-ton main line units.

Compressed air, supplied by a central surface compressor station located above the town, provides all requirements for both Phillipson and Storke levels. The five units that compose the compressor station—three 800-hp, one 600-hp, and one 500-hp—are driven by line start synchronous motors supplied by a 4000-kw substation.

Surface Plants

Crusher and mill sections represent a load of 22,745 hp with 1220 motors, ranging from 1 to 600 hp, utilized in the various beneficiation processes.

Crushers are supplied with power by a 3000-kw substation. Crusher control components are housed in a building remote from the crusher structures and push-button stations are located in the building for remote control of the starters for the operating units. Intricate interlocking between conveyor belts and crusher units, resistance-sensitive electronic relays, and power tripping relays eliminate ore pile-ups and crusher overloading. Liberal use of synchronous motors on the Symons cone crushers helps to maintain a 96 pct power factor.

The Storke gyratory crusher, one of the largest in the world, is driven by a 500-hp wound-rotor induction motor. The crusher motor control circuit is interlocked with the oil pressure system so that in case of oil pressure failure the motor will be deenergized at a predetermined time. The pan-feeder and impact belt are driven by dc variable speed drives to prevent overloading of the belt transporting the ore to the coarse ore bin. The crushing and conveying system is interlocked with a tramp iron detector.

The secondary crushing circuit consists of a pan-feeder at the coarse ore bin, conveyor belt feeding the Symons crusher, and conveyor belts to the mills. The entire system is interlocked to prevent ore piling in case of belt stoppage. The conveyor belt drives range from 125 to 350 hp.

The nine ball mill units in the No. 1 and 2 mill sections are equipped with 450-hp synchronous motor drives. Each mill and flotation unit has its own control center with power supplied by two 3000-kw substations. The main oil circuit breakers are in two separate buildings remote from the mill structure, and circuits are brought into the mill units in underground conduit.

Mills No. 3 and 4 have six 600-hp wound-rotor induction motor drives. Each unit composed of a ball mill, flotation unit, classifier, pumps, and other auxiliary equipment is served by a separate 1000-kw substation and is provided with the latest in control centers.

The substation for the regrind plant has a capacity of 3000 kw. The main plant drives consist of seven 200-hp synchronous motors. In addition there are flotation cell, pump and classifier drives, and control centers in this plant similar to those in mills No. 1 and 2. Power is also supplied to two 125-ft thickeners and a new 175-ft thickener.

Water Supply

Water supply for ore processing and tailings disposal and domestic use presents a major problem. Since the Climax operations are situated on the Continental Divide and watershed is limited, reclamation and conservation of water are of great concern.

Mill water is pumped 4 miles from Robinson Lake by three 700-hp motor-driven pumps of 3000 gpm capacity each and one 250-hp pump rated at 1000 gpm. Pumping station power is supplied by a 2000-kw substation. Motors are started across the line and capacitors connected across the motor leads prevent low voltage dips. Surge suppressors and hydraulically operated values protect the system against heavy surges and pump reversal in case of power failure.

Supplying Power and TV Signals for the Townsite

The more than 500 dwelling units in the townsite are supplied by three substations, two of 300-kw capacity each, and one with 500-kw capacity. Power is distributed throughout the townsite at 440 v and stepped down to 110/220 v for domestic use. The majority of the dwellings are equipped with electric ranges and the hotel uses electric power for all cooking, baking, and refrigeration.

One of the unique features is the community TV system provided by the Climax Molybdenum Co. as a community service to its resident employes. A total of 555 outlets have been installed to serve the permanent residents of Climax.

Antennas and head-end equipment of the broad band type for simultaneous reception of four Denver channels are installed on McNamee Peak at 13,803-ft elevation. Signals are brought from the antenna to the townsite over 12,000 ft of K14 coaxial cable with four amplifiers to maintain signal strength at normal levels. More than 50,000 ft of RG11U coaxial cable is installed in the townsite area to provide the circuitry to the dwelling outlets. Amplifiers and automatic level controls at strategic points compensate for circuit attenuation and provide each outlet with 1000 μv signal strength.

Training and Supervisor Development

THE supervisor is the real training man at Climax. This is true, of course, in many industries today. But at Climax the supervisor's importance as an instructor in the right methods of performing the jobs under his direction, his persistence in following through to see that work is done properly, and his ability to get men to follow his direction because they want to rather than because he wants them to—all these factors take on a keener importance in hard rock mining than in most heavy industries. The unavoidable dangers of mining, however, are so well known and identifiable that safety, efficiency, and production are inseparable.

This *work trinity* is the guiding principle toward which all planned training at Climax is directed. It is an old axiom that the right way of doing any job is the safe way or, conversely, that the safe way is the right way, ultimately resulting in greatest production. In the past this principle has not been easy to get over to the workman. Pressure for peak production has hit Climax recurrently through the years just as it has other mines. Present pressure to meet production goals as the result of Government stockpiling and increased industrial demand is reminiscent of war days. This sense of urgency occasionally tempts the individual workman to take shortcuts that prove costly in terms of production, injuries, or general confusion.

Supervisor Development

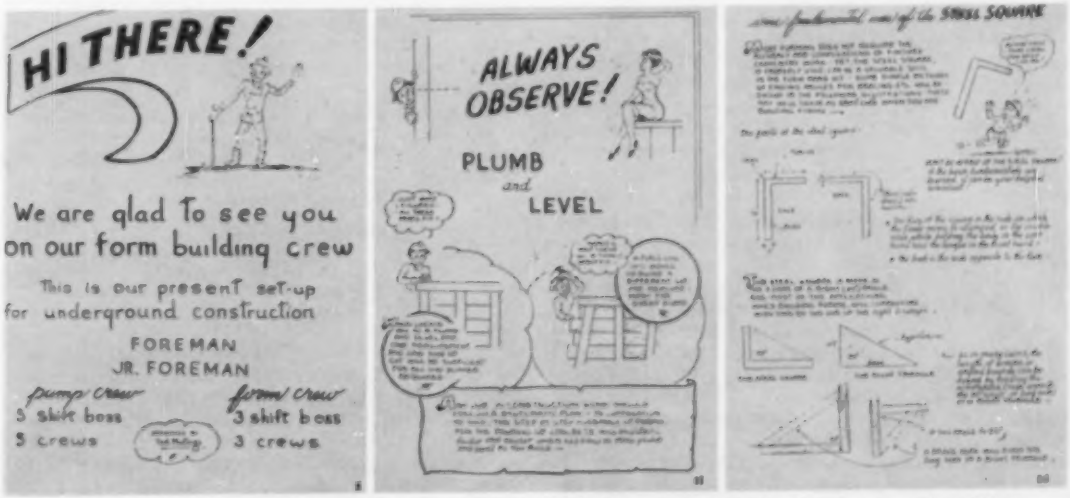
Realizing the importance of the supervisor, Climax has placed major emphasis on a supervisory development program. Since its creation two years ago, the objective of this program has been to make the operating supervisor a more efficient and effective leader and trainer of men. Management recognized from the outset that training was primarily a line responsibility rather than a staff function and that the main purpose of the training department should be to assist line executives and supervisors to fulfill their responsibilities in training subordinates. A building was set aside and equipped for training and safety meetings.

The basic training vehicle at Climax has been the conference method, which brings the full experience and considered thinking of operating officials to problems of training common to all supervisor personnel, namely, the need for 1. better understanding of the principles of leadership—of getting results through people, 2. greater skill in teaching the right methods of work, 3. increased ability to plan and simplify work methods, and 4. the consultative approach to operating and personnel problems as an effective and more frequently used management tool.

The supervisory development program has utilized the job relations, job methods, and job instructor training programs developed by the training within industry branch of the War Manpower Commission during World War II. These methods have since been widely used by industry and Government. The leadership program has also the *Man-to-Man on the Job* program of General Motors Corp.

The job instructor training has been given to some of the lead men as well as the supervisors in view of the fact that most of the job training is given by these key men rather than by the supervisor.

Conference leadership training has followed the pattern developed by the duPont Corp. in the smooth-functioning *discussion-leading* program. With the exception of orientation training given to employes new on a job and certain special courses which may introduce new equipment or work procedures, the fundamental basis of all Climax training is to develop the supervisor primarily through the conference method and then help clear the way for him to carry the training on down the line.



Three pages from a new training manual for the underground form-building crew. The booklet is pocket size and printed on washable and waterproof paper to stand mine service.

Looking west at the Outlaw and Calamity Mesa area from the Uncompahgre Swell. The La Sal Mountains of Utah are in the background. Orebodies in this district are a part of the Uravan mineral belt and lie about 60 miles southwest of Grand Junction.

Diversification Represented By Climax Uranium Co.

IN the spring of 1950, just two years after the inauguration of the domestic uranium program by the U. S. Atomic Energy Commission, the company entered the uranium mining and milling business through a newly formed subsidiary, Climax Uranium Co. This was the first venture into a new field of mining for Climax, which became the first old-time mining company, other than the vanadium producers, to become active in the embryo uranium industry.

Twenty-five years earlier, Arthur Bunker, president of Climax Molybdenum, organized and operated the U. S. Vanadium Co. at Rifle, Colo. With this background, he viewed the budding industry of the Colorado Plateau with much interest. Upon its formation, the new subsidiary entered into a contract with the U. S. Government and the potential uranium-vanadium properties of Minerals Engineering Co. were assigned to Climax Uranium Co. Funds for the construction of a processing plant and working capital were advanced by Climax Molybdenum Co. The old sugar mill building of the Holly Sugar Co. in Grand Junction was leased to house the extractive plant. Construction of facilities, which were based upon a new process developed for the treatment of both uranium and vanadium ores, was completed and the mill was placed in operation early in 1951. Subsequent to that time several alterations were made and the capacity of the plant increased. In terms of recoveries, the plant has proved to be one of the most efficient on the Colorado Plateau.

Recently the mill has been still further revised to increase capacity and simplify operations. A new crusher and sampling plant, completed this year, has been effective in streamlining ore purchasing and stockpiling, as well as providing feed for the mill. In addition, the percolation leach tanks for removal of the uranium from the slimes have been replaced with a modern continuous countercurrent leach plant.

Ores of the Colorado Plateau have been processed for many years, but uranium recovery is comparatively recent. Ores were first processed for radium and at one time the Plateau comprised the major world source of that element. After the African deposits were discovered the radium industry in the Plateau area could not compete economically. However, the vanadium content of the ores proved valuable and two companies built processing plants

that for many years recovered only the vanadium. While it was known that the ores contained uranium, there was no commercial market for any quantity of uranium, and little or no effort was made to recover the now critical element.

When the Government inaugurated the Manhattan Project, it was imperative that large amounts of uranium be obtained quickly from all possible sources. The mills processing Plateau ores for vanadium quickly converted so that uranium could be recovered as well. When Climax Uranium Co. began its construction in 1950, it was the first time that a plant had been initially designed for the recovery of uranium and vanadium from Plateau ores and erected with funds supplied by private capital.

Climax Uranium's mill utilizes a sand-slime separation method applicable to the majority of uranium ores in the Colorado Plateau. The general flowsheet of the mill was selected after careful consideration of known methods of beneficiating uranium and vanadium ores. The process is flexible as to ores that can be handled. Its use of waste acids in the neutralization step permits treatment of 10 to 15 pct lime ores.

Through the desirable features of sand-slime method, the mill is able to discard about 60 to 70 pct of the ore with minimum treatment. Effort can then be centered on the high grade slime product with a reduction in cost and improvement in recovery.

Development of any process is usually based on fundamental data obtained from laboratory results, pilot plant operation, and from general experience in other plant operations. Normally in ore processing the fundamental data will include characteristics of the ore to be treated. In the Colorado Plateau where deposits are generally small and vary widely in basic composition, it is impossible to determine the characteristics of the feed ore. Climax treats ore from 50 to 75 different sources, and the same is true of most of the other processing plants. It is for this reason that most of the processing plants in the Colorado Plateau are in a more or less continual state of change in an attempt to keep up with changing conditions of mill feed.

Since the ores are usually sedimentary sandstones they are easy to grind. The values of both uranium and vanadium are cemented upon the sand grains and in grinding the ore, the cemented values are in a large part broken loose as an ultrafine slime. This property can be both beneficial and detrimental.

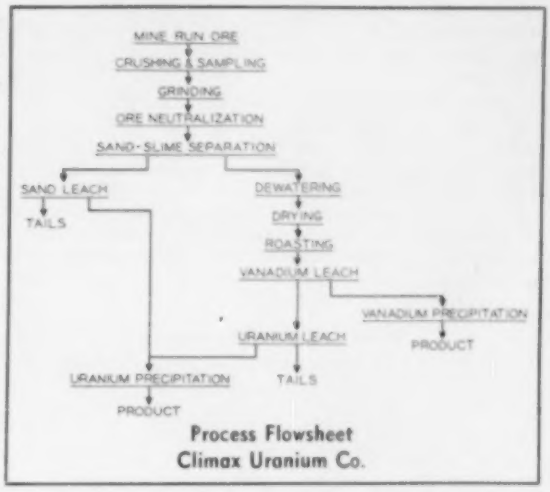
The neutralization section is an innovation introduced by the Climax Uranium Co. so that high lime ores may be handled. In the past high lime ores could not be processed in acid leach circuit for two main reasons:

1. Ores containing in excess of 4 to 6 pct calcium carbonate would, upon salt roasting, convert the vanadium into a water-insoluble calcium vanadate and vanadium recovery would be seriously affected.

2. Sulphuric acid used to leach the uranium would react with the calcium carbonate to form calcium sulphate, which in one form is plaster of Paris. The plaster of Paris would then set up in the now discarded leach tanks, stopping any percolation of the acid.

High Lime Ore No Problem with Ore Neutralization

In the Climax process, waste acids from the roaster gas scrubbers are used to neutralize the calcium carbonate contained in the ore. These waste acids are a mixture of hydrochloric and sulphuric acid. When calcium carbonate reacts with hydrochloric acid, it forms soluble calcium chloride that can be eliminated in the thickener overflow. The calcium carbonate that reacts with the sulphuric acid forms calcium sulphate which is quite insoluble in water and will, of course, be carried along with the ore. It has been found, however, that calcium sulphate upon roasting does not react with the vanadium to form calcium vanadate so that the conversion of the vanadium to water-soluble sodium vanadate occurs normally in the roaster. The calcium sulphate, also, is converted in the roaster to a dead-burned gypsum which does not set up. By use of this step, difficulties generally encountered with high lime ores can be eliminated.



Process Flowsheet
Climax Uranium Co.

At Climax Uranium ore is received from the crushing and sampling dept. at −1 in. and ground in a 4x6-ft Marcy rod mill in closed circuit with an Akins classifier. Neutralization is carried out by pumping the slurry from the Akins classifier overflow to one of five 8x8-ft agitator tanks where it is mixed with the waste acids at 1.5 pH. The five agitator tanks are operated in series to provide sufficient contact time for the reaction between the lime and acid to occur. The overflow from the No. 5 acid agitator goes to another agitator tank where NH_4OH is added to bring the pH up to 6. This step is taken to precipitate any uranium or vanadium that may have been dissolved in the acid neutralization treatment.

Other benefits that are obtained besides the neutralization of the lime in the ore are:

1. Overall acid consumption in the plant is reduced; rather than add new acid, waste acids can be utilized.
2. Plant make-up liquors containing small amounts of uranium and vanadium are sent through the ore neutralization where the uranium and vanadium are precipitated and recovered in subsequent operation. This enables plant control of pregnant liquors at optimum levels for ease in refining.

Another innovation in the Climax flowsheet is sand-slime separation. After the ore has gone through the neutralization step, it is pumped to a Dorr hydraulic sizer where a sand-slime separation is made. It has been found that by making this separation, 70 to 80 pct of the values contained in the

ore can be concentrated into the slime fraction which will constitute about a third of the weight of the original ore. The separation is made at about 150 mesh.

The +150 mesh fraction from the sizer is sent to a Dorr rake classifier where it is dewatered to 75 pct solids. The solids are brought in contact with a dilute sulphuric acid solution in percolation leach tanks where the uranium and 15 to 20 pct of the vanadium remaining with the sands are leached out. This liquor goes to the uranium refining section. The leach tanks percolate rapidly and produce uniformly good tailings.

Slime Filtration Problems

Slimes in the Colorado Plateau ores, whether mixed with the sands or handled separately, constitute one of the most serious problems facing the processors today. The ores in general contain appreciable amounts of claylike materials that are extremely difficult to thicken and to filter.

Climax has installed two submerged roto-disk filters to overcome this difficulty. The overflow from the hydraulic sizer contains about 6 to 8 pct solids. This dilute slurry is pumped to the submerged filters to thicken the pulp to 40 pct solids, the overflow from which goes to conventional disk filters, where the pulp is further dewatered to 50 to 55 pct solids. The filtrate from the submerged filters is returned as make-up water to the rod mill.

The submerged filters are so arranged that any of the sizer overflow that they cannot handle is sent to a 50-ft Dorr thickener where the material is thickened to about 20 pct solids. The underflow of the thickener is pumped to the disk filters for further dewatering.

It is felt that 1000 sq ft of filter area in the submerged filters will replace a 65-ft thickener. In fact they do a better job in that 20 to 25 pct solids is the maximum that can be obtained in the thickener underflow whereas the submerged filters can produce 40 to 45 pct solids. However, filters are more expensive to operate. Therefore, much experimental work has been done with various flocculating agents in an attempt to improve the thickening and filtering characteristics of the ores. Carboxymethyl cellulose gives the best results so far.

Thickener overflow is discharged into a series of settling ponds used as scavengers to guarantee against possible slime loss. Slimes carry a major portion of values, so losses at this stage are critical.

Salt Roast

Cake discharged from the disk filters goes in a screw conveyor to a salt mix agitator. Here the slimes are mixed wet with 12 to 15 pct by weight of salt. The salt is added to supply sodium ions which react with the vanadium in the ore upon roasting to form water-soluble sodium vanadate. After the salt addition, slimes are mixed in a pug mill with already dried slime diverted from the drier discharge in order to lower moisture content to 25 pct before the mix goes to the roto-louvre drier.

The drier discharge is conveyed to a ten-hearth, 20-ft diam Skinner roaster where the ore is roasted for 2 hr at 825°C. Two major reactions occur in the roast:

1. The major part of the vanadium compounds are converted to water-soluble sodium vanadate.

$$V_2O_5 + 2\ NaCl + H_2O \rightarrow 2\ NaVO_3 + 2\ HCl\uparrow$$

2. When the salt reacts with the vanadium and other constituents in the ore, it produces hydrochloric acid gas. This is recovered by passing the roaster gases through a scrubbing system and the acid thus made is used to neutralize the lime in the ore.

The calcine is discharged at 825°C from the roaster into a Baker cooler, which is a three-compartmented tube partially submerged in water. The calcine is cooled and discharged to a belt conveyor that delivers the calcine to the vanadium water leach.

The calcine is discharged into a small ball mill and ground with hot water. Very little grinding is done in this mill since only the particles larger than ¼ in. need be broken up. The slurry from the ball mill is discharged to a 6x6-ft agitator surge tank and then to a top-loading drum filter.

Leaching and Refining

The filtrate from the drum filter contains most of the water-soluble vanadium of the calcine. The cake is washed on the filter with hot water sprays and then is discharged to another agitator tank where it is repulped with hot water and is then filtered and washed on a second drum filter.

Solids from the second filter are pumped to thickeners for a final washing. The overflow is returned to the vanadium leach circuit, and the thickened pulp is sent to the new countercurrent uranium leach plant. In the leach plant, the uranium-bearing slime is mixed with sulphuric acid, then filtered on drum filters. This process is repeated four times under carefully controlled pulp densities and pH to produce maximum recovery of uranium in the ore.

The vanadium pregnant liquor from the drum filters is first clarified in pressure filters to remove any trace of sand that might have gone through the screens of the drum filters. The liquor is then pumped to a large agitator tank where it is heated to 90° to 95°C and sulphuric acid is added to bring the pH to 3.0. The vanadium will then precipitate as complex sodium polyvanadate $2Na_2O \cdot 7\ V_2O_5$, commonly known as red-cake. The precipitated liquor is placed in filter-bottom tanks where the red-cake is filtered, and the filtrate is sent to waste. The cake is thoroughly washed and is then charged into a fusion furnace where it is melted to form a dense black vanadium product.

The acid liquors from both the sand and slime leaching operations are sent to the uranium section where the uranium is removed and refined. Waste acid from this section containing residual acids and some vanadium is returned to the lime neutralization section.

Much effort, time, and money have been expended by the Climax Uranium Co. in its research activities. The processing of uranium is a relatively new field in the mining industry and while existing methods are fulfilling Government needs, the industry feels that many improvements can be made. The research dept. is conducting test work in its own laboratories as well as studying the results of the AEC research program. Many features that are being incorporated in the new mill recently completed in Grand Junction are the results of this research program. Until more is known of uranium processing, the research activities must continue at a vigorous pace.

Establishing Soaking Pit Schedules from Mill Loads

by R. D. Hindson and J. Sibakin

In order to devise a practicable soaking pit schedule for use at The Steel Co. of Canada Ltd.'s Hamilton Works, soaking pit heating temperatures, soaking times, pit capacity, and safe maximum mill drafts were correlated with fluctuations in the current or load of the bloom mill driving motor. Other variables such as total delays in the pit, rolling schedules, mill delays, and track times were also investigated.

IN order to show an easily applied and accurate means of establishing soaking pit heating temperatures, soaking times, pit capacity, and safe maximum mill drafts, these various factors are correlated herein with fluctuations in the current or load of the bloom mill driving motor.

Rolling practices have a considerable influence on the production capacity of a blooming mill. The maximum values of the torque, in particular, are of importance, since even instantaneous current peaks lead to the tripping of the motor by the overload relay and result in loss of mill time. The establishment of safe maximum drafts and accelerations for ingots of different sizes and of a soaking pit practice which would ensure a consistent and satisfactory plasticity of the metal is of considerable importance for increasing the efficiency of both the blooming mill and the soaking pits.

The Bloom Mill Dept. of the Hamilton Works, The Steel Co. of Canada Ltd., is equipped with one 44 in. mill driven by a 7000 hp motor with the setting of the overload relay at 22.0 ka. The speed of rotation of the motor is regulated after the Ward-Leonard system. There are three basic speeds of 9.5, 28, and 47 rpm and a further possibility of increasing the speed by weakening the field. This last possibility is hardly ever used during practical operations. The rolling program of the blooming mill is varied, both in the size of the ingots to be handled and in the steel grades. The total tonnage handled by the mill is about 2,000,000 ingot tons per year.

At the time of the investigation, the Bloom Mill Dept. was equipped with 22 soaking pits (6 regenerative, 14 bottom-fired, and 2 one-way top-fired pits) with a total bottom area of 2770 sq ft. The pits are fired with a blast furnace-coke oven gas mixture having a calorific value of 155 Btu per cu ft.

The foregoing figures show that the production program was such as to impose the necessity of a most efficient usage of the available equipment. For this purpose, the operations of the 44 in. mill and of the soaking pits were investigated, and the results of the investigation were used as a basis for a revised soaking pit schedule and drafting practice.

The plasticity of an ingot of a certain chemical composition when being rolled is determined mainly by the following factors: 1—the ingot size, both thickness and width; 2—the length of the gas soak; and 3—the surface temperature. The first two factors determine the uniformity of the temperature distribution over the cross-section of an ingot. The third factor introduces the level of the heating of an ingot.

The torque produced by an ingot being rolled is determined by the area of the metal displaced, its plasticity, and acceleration values. On the other hand, with shunt motors the torque is determined by the current. This can be assumed to be correct with only a small degree of error for compound motors with a relatively small effect of the series windings as long as the velocity is not regulated by weakening the field. Since the spread is relatively unimportant when compared to the width of an ingot and since it is also reduced several times during rolling by edging passes, the draft alone and not the area of the metal displaced may be taken into consideration with ingots of a similar size.

It is therefore possible to determine the main features of the heating and drafting of an ingot by measuring the current and acceleration of the mill motor. After the acceleration has been taken into account, the amount of current will be an indication of how the motor responds to a heating and/or drafting practice and these practices can be adjusted in order to get the desired result.

As peak currents are more likely when heavier ingots are rolled, the rolling of plate and slab ingots was investigated. Conditions prevailing when smaller ingots are rolled can be deduced from the results obtained on heavier ingots. All measurements were made when plain carbon grades under 0.15 pct C were rolled.

The motor current, the voltage across the armature, and the rpm were recorded simultaneously on synchronized charts, Fig. 1, which moved with the speed of 6 in. per min. Each draft was recorded by a special observer.

The rpm curve made it possible to establish the acceleration at any given moment. For purposes of correlation, the maximum current during a pass and the corresponding acceleration were used. The charts made it possible to establish the position of the roller's lever at any given moment as well as the total time of a pass.

The slab ingots were divided into three groups (28x35, 28x45, and 27½x53 in. ingots) and each group was investigated separately. Since they account for most of the current peaks, only flat passes were used for purposes of correlation, a total of 1373 having been investigated.

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The Transactions papers appearing in the Journal of Metals during 1955 will subsequently appear in AIME Transactions Volume 203, and may be permanently referenced to that volume.

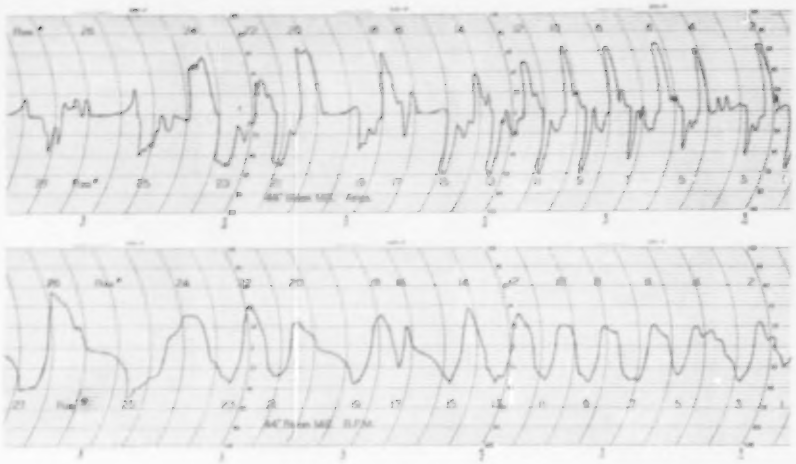

Fig. 1—Motor current, the voltage across the armature, and the rpm were recorded simultaneously on these synchronized charts. The charts were moved at a speed of 6 in. per min and each draft was recorded by a special observer.

The data were treated on the basis of multiple correlations. The current drawn by the motor was found to be almost independent of the velocity of rotation, as should have been expected. The influence of the acceleration while rolling under practical conditions was established and eliminated in cases when its magnitude was above the practical figure that corresponded to a normal rolling practice. As a result, the current could have been considered as a measure of only the drafts and properties of the ingot to a considerable extent. Thus, the experiment was held on a normal practical level and can be considered as representative of average conditions.

By using the same technique, the influence of the acceleration can be removed completely and practical conditions simulated by assuming a certain admissible value of the acceleration.

The surface temperature of the ingots was established experimentally and changes with the carbon content of the steel, roughly following the pattern of the solidus line of the Fe-C diagram. It is 2325° to 2350°F for carbon contents of up to 0.15 pct. This is the temperature of the waste gases as measured by a thermocouple in the outlet, 6 in. from the face of the wall in the three or four bottom corners of a pit. This figure is not necessarily the true temperature of the ingot surface but has a reliable relative value when all measurements are done in the same way. This temperature is obviously dependent on the way and place where it is measured and its absolute value may not be the same with different plants and pits.

The influence of the soaking temperature was investigated on well soaked 28x45 in. ingots (at least 2 and up to 3½ hr gas soak) within a range of 2340° to 2370°F. No increase in the plasticity of the metal when the temperature was raised from 2340° to 2370°F was detected. Although the temperature range was quite narrow, a change in plasticity at this temperature level will probably be noticeable only if the temperature is lowered by at least 50°F. The plasticity, however, becomes more sensitive to temperature changes at lower levels. Thus a soaking temperature of 2325° to 2350°F for low carbon grades is sufficient and a further increase does not lead to a reduction of the mill motor current at similar drafts.

The influence of the gas soak, i.e., time at temperature, on the mill motor current was investigated on ingots of different sizes which were soaked within the required temperature range of 2325° to 2350°F. The length of the gas soak was varied between 40 and 220 min and the greater majority of the ingots had a track time of 2 to 6 hr. The drafts were correlated with the maximum current during a pass after the extreme values of acceleration were eliminated. The ingots were divided into groups with similar gas soaks (within 10 min) and each group was treated separately.

The result of such a correlation for 27½x53 in. ingots is shown in Fig. 2.

The two lower lines are relatively quite close to each other, the difference being within the limit of an experimental error, and can be combined. This means that there is no change in the plasticity of an ingot of this size under practice conditions when the length of gas soak is increased from 95 to 160 min.

Fig. 3 shows the relative position of the lines relating the drafts with the maximum mill motor current after the lines for 95 and 160 min gas soak had been combined. Both figures indicate that a gas soak of 90 min should be selected for 27½x53 in. ingots.

The same correlations for different lengths of gas soak were made for 28x35 and 28x45 in. ingots. All other conditions being approximately equal, a gas soak of about 50 min when compared with longer soaking times has shown no change for 28x35 in. ingots and the beginning of a tendency for stiffening for 28x45 in. ingots. As was shown before, 27½x53

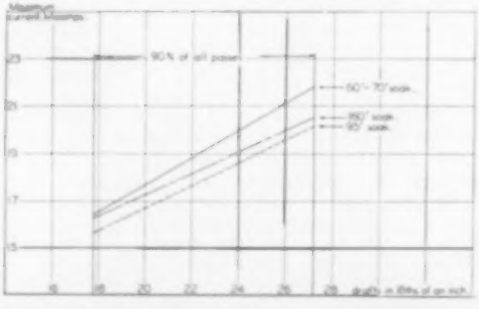

Fig. 2—Influence of the gas soak, i.e., time at temperature, on the mill motor current was investigated for 27½x53 in. ingots. The ingots were soaked within the required temperature range of 2325° to 2350°F. The gas soak was varied in length from 40 to 220 min and the drafts were correlated with the maximum current during a pass after the extreme values of acceleration were eliminated.

on each pass would be desirable. A draft recorder

22x24, 23x25, 24x26 in.; 2—small slab ingots: 28x35

in. ingots had under those conditions a rather pronounced tendency for stiffening. It follows that a satisfactory degree of plasticity will be obtained after the following minimum gas soak: 45 min for 28x35 in. ingots, 60 min for 28x45 in. ingots, and 90 min for 27½x53 in. ingots.

The lines on Figs. 2 and 3 are drawn through the average values of the current for each draft and the variance is distributed equally on both sides of the lines. The value of the standard deviation of the current when combined with the average lines as shown on Figs. 2 and 3 allows establishment of a draft at which the maximum allowable current (corresponding to the setting of the overload relay) will be exceeded in only a certain definite number of cases. This level of safety was selected to be 10 pct, and the corresponding draft is the maximum safe draft at which the maximum current of 22.0 ka will be exceeded in not more than 10 pct of all cases.

These maximum safe drafts were found to be: 1 7/16 in. for 27½x53 in. ingots, 1⅝ in. for 28x45 in. ingots, and 2 in. for 28x35 in. ingots.

As the levels of probability were established with only the extreme cases of acceleration excluded, they are representative of normal practice without restrictions. The maximum draft can be further increased within the same safety limit if high acceleration figures during rolling are avoided. The practical figures for the percentage of all flat passes which required a current of 22.0 ka (or 23.0) and over at different drafts were tabulated as a check and the resulting curve for 28x45 in. ingots is shown in Fig. 4. The frequency distribution based on practical figures shows that at the suggested maximum safe draft the magnitude of the current is well within the selected safety limit.

In order to check the influence of the drafting practice on the rolling time of an ingot down to the same slab size, the overall rolling time was established. Frequency distributions of drafts for all ingots rolled below and above the average rolling time were then drawn. The results for 28x45 in. ingots are shown in Fig. 5. The ingots with a short rolling time had a considerably more consistent schedule of drafts, half of all flat passes lying within ¼ in. from the upper safe limit of 1⅝ in. Inconsistent erratic drafting resulted in a loss of approximately 0.4 min on each ingot.

Light drafts result in a time loss by increasing the number of passes; too heavy drafts result in time losses by increasing the number of trippings of the motor. The influence of the draft on the time for a flat pass is shown in Fig. 6 and supports the foregoing statement.

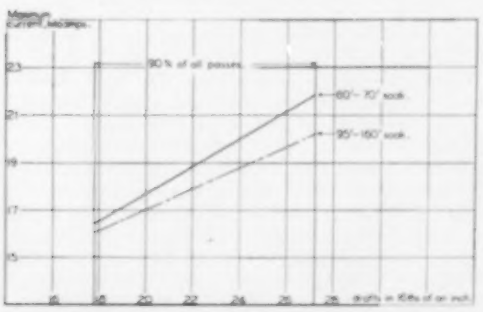

Fig. 3—Lines relating the drafts with the maximum mill motor current are shown after lines for 95 and 160 min gas soak had been combined; see Fig. 2. Data shown are for 27½x53 in. ingots. Selection of a 90 min gas soak is indicated.

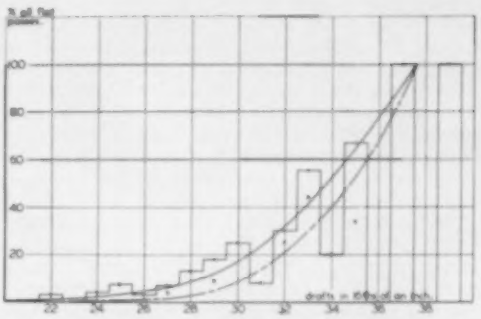

Fig. 4—Practical figures for the percentage of all flat passes which required a current of 22.0 or 23.0 ka and over at different drafts were tabulated and the resulting curve for 28x45 in. ingots is shown. The solid line represents a current of >22.0 ka and the broken line that of >23.0 ka.

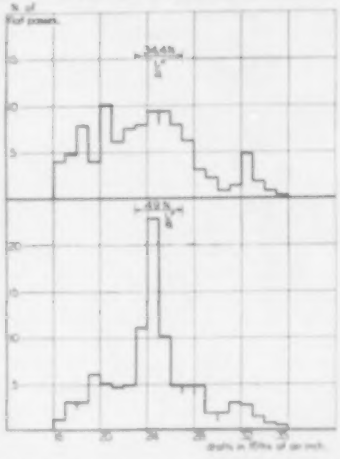

Fig. 5—The influence of the drafting practice on the rolling time of an ingot down to the same slab size was checked by establishing the overall rolling time. Frequency distributions of drafts are shown for 28x45 in. ingots rolled below and above the average rolling time. Top section of the graph shows the distribution for an average rolling time of 3.2 min; bottom section is for an average rolling time of 2.8 min.

Thus a consistent drafting practice at or about the upper safe limit for a draft with a minimum acceleration with steel between the rolls (higher speed of entry) is conducive to a high production level. The upper safe limit of a draft on a flat pass may be increased if acceleration with steel between the rolls is avoided or if the overload relay is set on a higher peak current.

It would appear from the foregoing that some type of measuring instrument to record the drafts taken

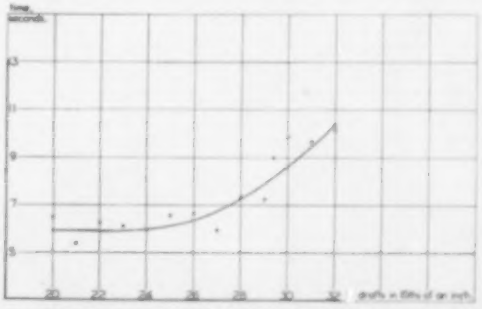

Fig. 6—Influence of the drafts on the time for a flat pass is shown for 28x45 in. ingots. The curve supports the contention that light drafts result in a time loss by increasing the number of passes. Conversely, too heavy drafts result in time losses by increasing the number of trippings of the motor.

bottom-fired Amco pits in June 1953, the percent-

practice and indicate only the ultimate capacity of

on each pass would be desirable. A draft recorder has been designed by the Electrical Dept. for this purpose. It utilizes a Sanborn recorder working with four charts and is designed to measure the opening between the rolls, the actual draft taken, and the mill motor current. The fourth chart can be used to measure the rpm of the mill motor if required. A timing pen which marks each second on the chart allows the accurate establishment of the rolling time for each pass and each ingot.

The advantages of such a draft recorder should be readily apparent. It will provide closer control of rolling practices, allow a more accurate establishment of drafting schedules, and enable more exact measurements to be taken for experimental work. The first two advantages mentioned should also result in an increased production rate in the mill.

Table I. Number of Ingots Charged into Bottom-Fired Amco Pit

Type of Ingot	No. of Ingots Charged
22x24	16
23x25	12
24x26	12
28x35	8
28x39	8
28x45	6
28x48	6
27½x53	6
26x53	6

The soaking pit schedule should be developed in such a way that the time soak (the minimum time in pits) would include the length of the gas soak required for a satisfactory plasticity of the ingot at the required temperature. A specified time soak in addition to a gas soak is necessary to cover abnormal cases such as failure of recording equipment, prevention of rolling green steel, etc.

As heating conditions may be different for different pit constructions, bottom-fired, top-fired, and regenerative pits were investigated separately. The conditions and results of the investigation for bottom-fired pits will be discussed in detail as representative of the investigation technique in all cases.

Since reliable information on temperature and start and finish gas soak was of considerable importance, the instrumentation of the pits was inspected first and all defects were eliminated. Pits that were unquestionably satisfactory as to the uniformity of the heating, quality of the gas soak, and its determination were used for collecting experimental data.

The charges were subdivided into four groups according to the size of the ingots: 1—small ingots: 22x24, 23x25, 24x26 in.; 2—small slab ingots: 28x35 in.; 3—medium slab ingots: 28x45 in., and 4—heavy slab ingots: 26x53 and 27½x53 in.

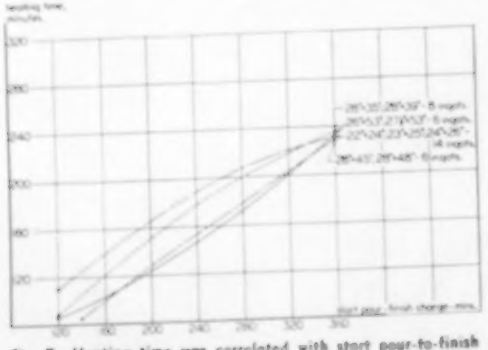

Fig. 7—Heating time was correlated with start pour-to-finish charge time for charges made in Amsler-Morton (bottom-fired) pits with standard numbers of ingots.

Each group was treated separately and the similarity of the ingot sizes in each group justified the assumption that the heating process within each group is practically similar.

The heating time (time from finish charge to start gas soak) was correlated with the number of ingots charged, the start pour-to-finish charge time, and the temperature of the gas soak for each ingot size group separately. The consideration of the heating time and not of the total time makes the problem independent of the length of the gas soak, which is different for the various ingot sizes.

The problem was treated as a multiple correlation, curvilinear for the start pour-to-finish charge time in order to bring this most important factor as close as possible to practical conditions. The influence of the different factors on the heating time was separated and the results correlated for a charge of a certain weight or number of ingots and a temperature of 2325° to 2350°F.

It has been established in practice that, allowing for a normal distribution of ingots in the pit and the possibility of handling them with a crane, the number of ingots given in Table I can be charged into the bottom-fired Amco pit with an available hearth area of 142.5 sq ft and a burner 5 ft diam.

It was not possible to find a significant difference in temperature in the corners of the pit for charges which had 10 to 16 small ingots. The percentage of the bottom area of the pit covered by the charge is around 40 in all cases. This seems to indicate that these charges are at their best values and can be heated up quite satisfactorily. For purposes of calculation, the number of small ingots in a charge has been assumed to be 14, the average between 12 and 16.

Table II. Relationship for Estimating Time Soak as Applied to Charges Heated in Amco Pits in June 1953, Percentage of All Charges Covered

Type of Ingots, In.	Pct of All Charges Covered
22x24, 23x25, 24x26	81.7
28x35, 28x39	85.1
28x45, 28x48	87.1
26x53, 27½x53	79.9

The relationship between the heating time and the start pour-to-finish charge time for the standard charges was established first, Fig. 7. The minimum length of the gas soak has been established in the earlier section of this paper. The time soak (minimum time in pits) will be obtained when the length of the gas soak is added to the heating time. The resulting relationship between the start pour-to-finish charge time and the time soak for different ingot sizes is shown in Fig. 8. The relative position of the lines allows an estimation of the time soak in a similar way for ingots of all sizes. The suggested relationship is valid for all cases. The slope of the suggested line as shown in Fig. 8 is not the best possible fit to the practical lines but the best fit of a simple expression for the suggested relationship to practical conditions. The suggested relationship can be expressed in the following way: the time soak is equal to half the start pour-to-finish charge time plus 2 hr.

When the suggested relationship for estimating the time soak was applied to all charges heated in

bottom-fired Amco pits in June 1953, the percentage of all charges covered by it was as is shown in Table II.

Table II indicates that the relationship between time soak and start pour-to-finish charge time as suggested in its simple form is satisfactory from the practical point of view, but the relationship does not cover the extreme cases which are due to some abnormalities in heating or pit conditions and which cannot be normally anticipated. When only extreme cases are taken into consideration (allowing for a reasonable inaccuracy in the time reported by the heater and observer of about 5 min), the percentage of all charges covered will be at least 90.

The relationship "half start pour-to-finish charge time plus 2 hr" for the normal standard charges (Table I) and highest temperature range of 2325° to 2350°F covers almost all practical conditions.

As the time soak includes the normally required gas soak in 80 to 90 pct of all cases, there will be some charges when the gas soak will not be completed within the estimated minimum time soak. In order to ensure the readiness of the steel for rolling, a pit should be drawn only when both the conditions of a minimum time soak and minimum required gas soak are fulfilled.

The same reasoning and experimental technique were applied to the top-fired and regenerative pits and resulted in the relationship between the time soak and transfer time as shown in Table III. The suggested time soak includes the normally required gas soak in 80 to 90 pct of all cases. The relationship is simple, uniform for pits of different construction, and brings the soaking pit schedule on a more realistic basis.

The minimum time soak becomes a measure of the heating ability of different pits and can be used for the estimation of the pit capacity.

The time soak as established previously is the minimum time from finish charge to start draw. The time from start draw to finish charge is part of one full heating cycle and is dependent on the mechanical equipment of the shop, location of the pit relative to the mill, size of ingots, etc. It has to be established experimentally for each particular shop. In this case it was established to be 1 hr for the regenerative and bottom-fired pits and 1¼ hr for the top-fired pits.

Table III. Relationship Between the Time Soak and Transfer Time for Top-Fired, Amco, and Regenerative Pits

Type of Pits	Minimum Soak Time
Amco, bottom-fired	Half start pour-to-finish charge time + 2 hr
Regenerative	Half start pour-to-finish charge time + 2¼ hr
One-way top-fired	Half start pour-to-finish charge time + 3 hr

The estimation of the pit capacity for bottom-fired pits only will be discussed in detail, since it is representative of the technique used for other pits. The minimum possible time of one full heating cycle for bottom-fired pits under conditions prevailing in the plant will be equal to time soak plus 1 hr. This figure is not applicable to each individual charge but is valid on a long term basis. The number of charges per day and per pit and the turnover of a charge (hours per charge) under this condition are shown in Table IV.

The figures in Table IV represent the maximum theoretical pit capacity when there are no delays whatsoever. These figures can never be attained in practice and indicate only the ultimate capacity of a pit. However, these figures permit assessment of the influence of different delays on the pit capacity independently from other variables.

In order to arrive at practically attainable capacity figures, various delays in the operations of a pit have to be estimated. One of the unavoidable delays is the time when the pit is down for repairs. The time loss involved (including cooling, etc.) can be assumed to be 5 pct of the total time. This figure has been established over a period of several months for all pits. This delay is close to constant and cannot be changed considerably.

The second source of unavoidable delays is the time lost as a result of holding a charge in the pit over the time when it is ready to draw. These delays are due to several factors such as: 1—mill down for repairs, 2—mill not prepared or unable to roll a charge which is otherwise ready, 3—several pits being ready simultaneously, or 4—the rolling schedule not being sufficiently adapted to the soaking pit time table, etc. Although it is absolutely impossible to avoid these delays altogether, it may be possible to decrease them by a certain reasonable figure.

Table IV. Minimum Time of One Full Heating Cycle for Bottom-Fired Pits

Start Pour-to-Finish Charge Time, Min	Hr per Charge	No. of Charges per Day
120	4.00	6.00
150	4.25	5.65
180	4.50	5.33
210	4.75	5.05
240	5.00	4.80
270	5.25	4.57
300	5.50	4.36
330	5.75	4.17
360	6.00	4.00

In order to assess the magnitude of this delay, the time over finish gas and time soak for each charge and pit for the period of 56 consecutive days was established and an average per month and pit calculated. The average total delays in pits over time ready for bottom-fired pits under practical conditions were found to be 160 hr per month per pit.

This figure is equal to all the delays of charges in a pit and is representative of normal practice. It is obviously impossible to eliminate it altogether, but a reduction of 20 to 30 hr might be possible. The figure in itself is an indicator for the efficiency of the usage of the pit time for pits of similar construc-

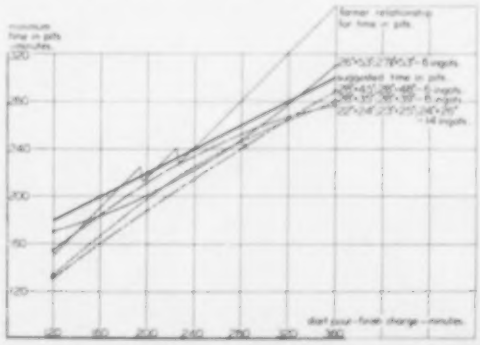

Fig. 8—To the data of Fig. 7 was added the length of the gas soak in order to obtain the time soak (minimum time in pits). Again, charges were made in Amsler-Morton (bottom-fired) pits with standard numbers of ingots.

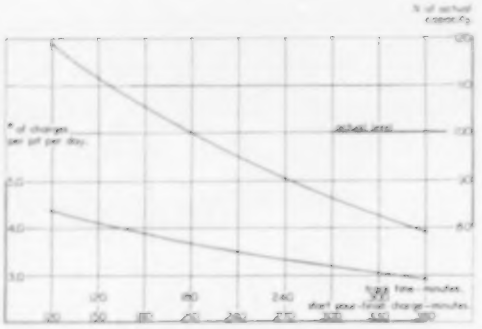

Fig. 9—Influence of start pour-to-finish charge and track times on the capacity of a pit under actual conditions was plotted for charges made in Amsler-Morton (bottom-fired) pits.

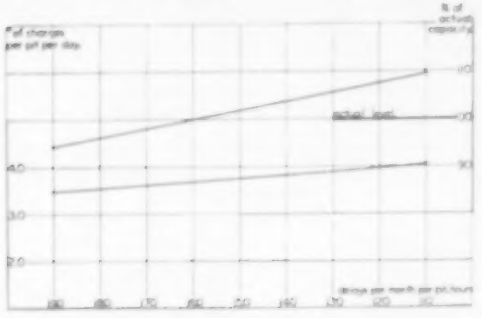

Fig. 10—Influence of delays on the capacity of a pit under actual conditions with a track time of 3 hr was determined for charges made in Amsler-Morton (bottom-fired) pits.

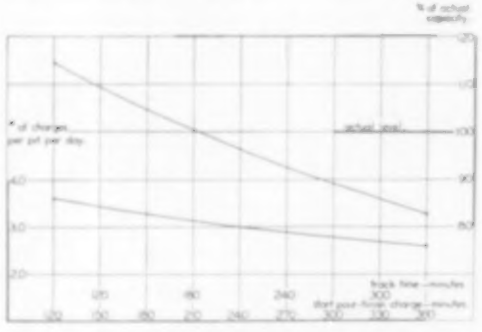

Fig. 11—Influence of start pour-to-finish charge and track times on the capacity of a pit under actual conditions was plotted for Nos. 23 and 24 one-way top-fired pits.

tion and can be used for assessing the influence of delays in the mill on the pit capacity.

Assuming a start pour-to-finish charge time of 3½ hr (conditions of June 1953), a corresponding turnover of 4¾ hr can be taken from Table IV. Hence the number of charges per day per pit will be equal to: $720-36-160/4\frac{3}{4}\times30 = 3.68$ and the turnover of a charge: $24/3.68 = 6.52$ hr. The numerator of the first fraction is the time available for straight heating cycles during one month and the denominator contains the turnover of a charge at a given transfer time with no delays. The figures in the numerator bring the problem to normal practical conditions and the fraction allows establishment of the influence of the transfer time and delays on the pit capacity separately.

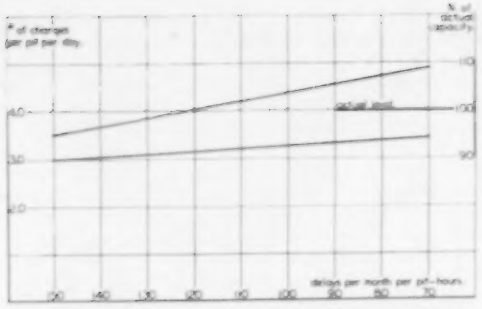

Fig. 12—Influence of delays on the capacity of a pit under actual conditions with a track time of 3 hr was determined for Nos. 23 and 24 one-way top-fired pits.

Table V. Pit Capacities of Bottom-Fired, Top-Fired, and Regenerative Pits

	Bottom-Fired Pit		Top-Fired Pit		Regenerative Pit	
	Charges Per Day	Turn-over Hr	Charges Per Day	Turn-over Hr	Charges Per Day	Turn-over Hr
Start pour-to-finish charge time, min, 120	4.37	5.49	3.58	6.70	3.82	6.28
150	4.11	5.84	3.42	7.02	3.63	6.61
180	3.88	6.18	3.27	7.34	3.45	6.96
210	3.68	6.52	3.13	7.67	3.30	7.27
240	3.49	6.88	3.01	7.97	3.15	7.62
270	3.33	7.21	2.89	8.30	3.02	7.95
300	3.18	7.55	2.79	8.60	2.90	8.28
330	3.04	7.89	2.69	8.93	2.79	8.60
360	2.91	8.25	2.59	9.27	2.69	8.92
Delays per month per pit, hr*						
190	3.47	6.92	—	—	—	—
180	3.54	6.78	—	—	—	—
170	3.61	6.65	—	—	3.12	7.69
160	3.68	6.52	—	—	3.18	7.55
150	3.75	6.40	2.97	6.06	3.24	7.41
140	3.82	6.28	3.02	7.95	3.30	7.27
130	3.89	6.17	3.08	7.79	3.36	7.16
120	3.96	6.06	3.13	7.67	3.42	7.02
110	4.03	5.86	3.18	7.52	3.48	6.90
100	—	—	3.24	7.41	3.54	6.78
80	—	—	3.30	7.27	3.60	6.67
80	—	—	3.36	7.14	—	—
70	—	—	3.41	7.04	—	—

* Figures in this half of the table were obtained at a constant transfer time of 3½ hr.

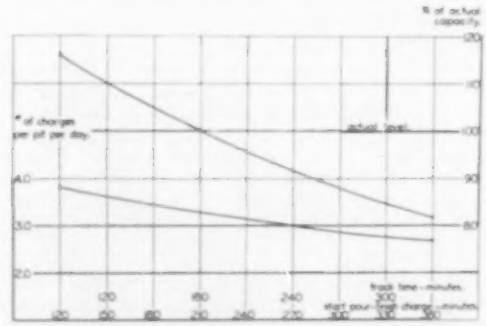

Fig. 13—Influence of start pour-to-finish charge and track times on the capacity of a pit under actual conditions was plotted for Nos. 3 through 8 regenerative pits.

The actual figures for the period from May 26 through June 30, 1953 were: total pit time for 14 pits, $24x36x14 = 12{,}096$ hr; total number of charges heated, 1,854; turnover of a charge, $12{,}096/1{,}854 = 6.52$ hr; and number of charges per day per pit, $24/6.52 = 3.68$.

The coincidence of practical and estimated figures obtained for similar conditions (transfer time, de-

Table VI. Soaking Pit Schedule Adopted on the Basis of Experimental Results

I. Heating Ranges—All Steels to be Heated Without Washing at Specified Temperatures

1. *Amcos and Old Pits Less Sulphur Grades*

C, Pct	Ranges, °F
0 to 15	2325 to 2350
16 to 30	2300 to 2325
31 to 50	2275 to 2300
51 to 80	2250 to 2275
81 plus	2225 to 2250

2. *Sulphur Grades*

Add 25° to the ranges given under No. 1.

II. Gas Soak

Size of Ingot	Gas Soak, Min*
22x24 BEU	45
23x35 BED	45
28x35 BED	45
28x45 BED	60
26x53 BED	90
All KHT slabs	120

* 1—When start pour-to-finish charge time is over 6 hr, add 15 min to the times given.

2—For regenerative and top-fired pits, add 15 min to the times given.

III. Time Soak and Minimum Time in Pits—For Additional Information, see Grade Book and Special Memoranda

Pit Type	Time Soak*
Bottom-fired	½ SP-to-FC† + 2 hr
Regenerative	½ SP-to-FC† + 2¼ hr
Top-fired	½ SP-to-FC† + 3 hr

* 1—To avoid green steel for short track times, minimum time soak must be 2½ hr for small ingots and 3½ hr for slab ingots.

2—If start pour-to-finish charge time is over 6 hr, use the specified gas soak schedule only.

† SP-to-FC represents start pour-to-finish charge time.

IV. Cooling Data—See Grade Book or Special List for Grades Requiring Cooling

SP-to-FC,* Hr	Pit Temperature at Charge, °F	Maximum Temperature in 2 Hr, °F
3 to 3½	1800	2100
3½ to 4	1700	2000
4 to 5	1600	1900
5 to 6	1500	1800
6 to 8	1300	1600
8 to 12	Black walls	Gas not to exceed 600 cfm for 1 hr, then gradually increased
12 and over	Black walls	Shut down for 45 min, then follow temperatures for 8 to 12 hr SP-to-FC

* SP-to-FC represents start pour-to-finish charge time.

lays) seems to indicate that all constants were established correctly and that the suggested approach can be used for the estimation of the influence of the transfer time and mill delays separately on the pit capacity under practical conditions.

The influence of the start pour-to-finish charge time and of the delays on the pit capacity is shown in Figs. 9 and 10. The figures of the graphs were calculated on the basis of the start pour-to-finish charge time. In order to switch to track time, it can be assumed that it is 30 min shorter for the Hamilton Works' conditions of operation.

The pit capacities of one-way top-fired and regenerative pits were estimated in the same way and the results obtained, which check closely with practical figures, are shown in Table V.

All figures in the second half of Table V were obtained at a constant transfer time of 3½ hr. The same relationships are shown in Figs. 11 through 14 in graphical form.

Using the tables or graphs, the capacity of one pit for different time periods as influenced by the track time and/or mill delays can be estimated. The influence of both variables is cumulative and can be summed when both undergo changes simultaneously. Their relative importance can be compared. The values obtained are close approximations to different practical conditions but should be considered as applicable to an average pit on a long term basis.

The figures for the number of charges per day per pit can be used as a basis for the assessment of the pit production capacity in tons. The average weight of a charge under normal operating conditions during the experimental period should be established for this purpose. The number of charges per day is to a certain degree independent of the average charge weight. The soaking pit schedule based on the results obtained is now in use and is shown in Table VI.

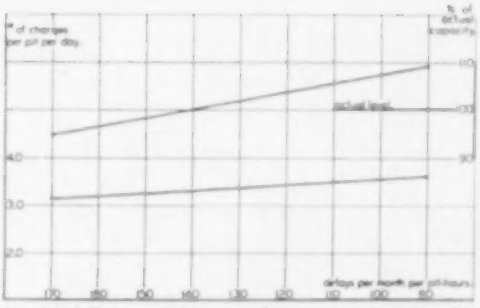

Fig. 14—Influence of delays on the capacity of a pit under actual conditions with a track time of 3 hr was determined for Nos. 3 through 8 regenerative pits.

Conclusions

The current of the bloom mill driving motor is affected mainly by the size of the draft. The acceleration of the motor within its usual limits when steel is between the rolls also influences the current but to a smaller extent.

A change of the temperature of the pits at the investigated level (2350°F) will probably be of influence only when it is at least 50°F.

The following minimum time for gas soak for a normally operating pit will ensure a consistent and satisfactory plasticity for rolling: for 28x35 in. ingots, 45 min; for 28x45 in. ingots, 60 min; and for 27½x53 in. ingots, 90 min.

Unnecessary standing of ingots on the line or in the buggy before rolling should be avoided, since even an additional 1 to 2 min above the average time has a tendency to decrease the overall plasticity of the ingot between the rolls.

The maximum safe drafts for the authors' conditions (7000 hp motor, 22.0 ka maximum) are: 2 in. for 28x35 in. ingots, 1⅝ in. for 28x45 in. ingots, and 1 7/16 in. for 27½x53 in. ingots. The upper safe

limit for a draft may be pushed up if unnecessary acceleration of the motor with steel between the rolls is avoided. Consistent drafting at the safe maximum limit is conducive to increased production rates.

The use of a drafting recorder has been suggested and should result in closer control of rolling practice and a more accurate establishment of drafting schedules which, in turn, could be expected to increase the production of the mill. The recorder will measure the mill opening, actual draft taken, and the mill motor current.

The rolling should be carried out at maximum speed, but minimum acceleration when steel is between the rolls should be avoided. A speed at biting rather close to the final speed of rolling will eliminate high values for acceleration at full load.

The thermocouple readings on the Amco pits are good indicators of the condition of the steel in the pits. Except in abnormal cases, their readings can be of considerable help to the pit personnel. Correct interpretation of the readings for different types of operation (pit shut-down, etc.) is required. Abnormal pit conditions, such as hot or cold corners, can be detected if thermocouple readings are followed systematically and eventually repaired. A graphical representation of couple readings before draw for one charge per shift and pit is desirable for this purpose. The thermocouples and controls require systematic attention and should be looked after by a repair man who contacts the heating foreman.

The minimum time soak for a gaseous fuel with 155 Btu per cu ft can be related to the transfer time as follows: for bottom-fired Amco pits, half start pour-to-finish charge time plus 2 hr; for one-way top-fired pits, half start pour-to-finish charge time plus 3 hr; and for regenerative pits, half start pour-to-finish charge time plus 2¾ hr. The suggested time soak can be applied to ingots of all sizes due to the peculiarity of heat losses and later reheating.

As the time soak, which includes the required gas soak, covers only 80 to 90 pct of all cases, there will be a few charges when the gas soak will not be completed within the required time soak. Therefore, a pit should be drawn only when both a minimum time soak and gas soak requirements are met.

The suggested time soak is the minimum time in pits necessary to meet the requirements of a satisfactory plasticity of an ingot for a normal practice and a 6-1 gas mixture. It can be used as a base for the estimation of the capacity of a pit. As the minimum time soak does not cover any delays in the pit, including the holding of a charge in the pit over finish soak for some unavoidable reason, the delays have to be estimated on the basis of an average practice.

The figure for the total delays in the pit is of considerable magnitude, but it is absolutely impossible to eliminate it completely. It can be reduced by avoiding mill delays and adapting the rolling schedule (drawing of pits) as far as possible to the pit schedule (time when pits will be ready for draw). The estimation of the time soak adapted to different pit constructions should give the mill foreman a better overall picture of when the pits are ready to roll.

The influence of the track time and delays on the production capacity of the pits was determined by each variable separately and their influence was found to be cumulative. The information can be used for future estimations of economic considerations.

The production capacity of different pit types in tons can be determined on the basis of the weights of an average charge. The correspondence between calculated and practical figures was good.

Pit capacity estimations are valid on a long term basis of at least one month.

All figures obtained for the pits are valid for a fuel of 155 Btu per cu ft. Should the calorific value of the fuel be different, the figures would have to be adjusted to the new heating conditions although the general trends would remain the same.

The practice of heating pits that are not fully charged up to the practically established standard (about 40 pct of the bottom surface covered by the charge) should be avoided, since it affects the production detrimentally. Overcharged pits should be avoided for the same reason and also for quality considerations.

This investigation confirmed a previous decision to purchase four new bottom-fired pits to handle a maximum tonnage of 2.2 million tons per year.

Acknowledgments

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Discussion of this paper, if any, will appear in JOURNAL OF METALS, November 1956, and in AIME Metals Branch *Transactions*, Vol. **206**, 1956.

Technical Note

High Nitrogen Cast Austenitic Steels

by John F. Carlson and Victor F. Zackay

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CURRENT experimental work on the development of gas turbine engines in the automobile industry has emphasized the need for a relatively low cost nonstrategic castable alloy for service in the 1200° to 1400°F temperature range. Ferritic steels

of the 12 pct Cr type are inadequate for service above 1100°F. Cobalt and nickel-base alloys, such as Haynes 31 and General Motors GMR-235, are excellent castable high temperature alloys but are costly and strategic in nature for large scale mass-produced applications.

A long range program is currently being conducted at the Scientific Laboratory of the Ford Motor Co. for development of heat-resistant materials of low strategic index. As a result of this program, a wrought nickel-free Cr-Mn austenitic steel has been developed for possible use at moderately elevated temperatures. The promising heat-resistant behavior of these wrought Cr-Mn-Mo-N steels led to an evaluation of their as-cast properties. The objective of the cast program was to evolve a castable Cr-Mn-Mo-N alloy having heat-resistant properties equivalent to those of wrought Timken 16-25-6 in the 1200° to 1400°F temperature range.

In general, cast austenitic iron-base alloys require greater proportions of austenite-forming elements than do wrought alloys of the same general composition. For this reason and for maximum retention of nitrogen, the manganese content was held to within the range of 16 to 18 pct. Adequate oxidation resistance in these nickel-free steels requires 14 to 16 pct Cr. The hot strengthening effect of nitrogen was aided by the addition of molybdenum. A base composition of 16 pct Cr, 18 pct Mn, 2 pct Mo, and 0.5 pct N was selected.

The austenitic structure in iron-base Cr-Mn-Mo-N alloys is metastably retained by rapid cooling from elevated temperatures. This nonequilibrium phase tends to decompose when subjected to the combined action of thermal and mechanical forces. The degree of austenitic stability in any given environment is in turn closely dependent upon the relative proportion of the α or γ-forming elements in the alloy. Thus the guiding principle in the alloy chemistry of these steels is to achieve the greatest austenitic stability over the desired temperature range consistent with the hot strength requirements of the particular service application.

The creep-rupture characteristics of the base composition 16 pct Cr, 18 pct Mn, 2 pct Mo, and ½ pct N are shown in Fig. 1, on a Larson-Miller plot, where

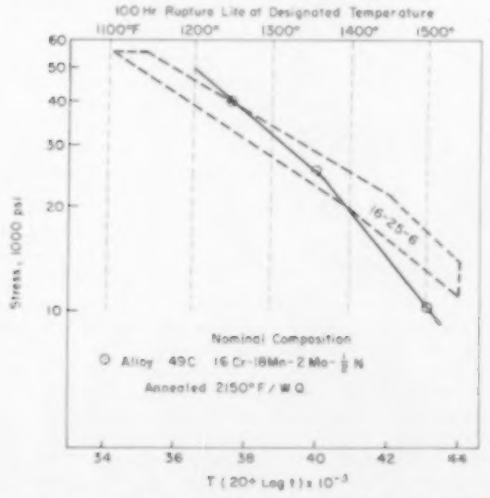

Fig. 1—Creep-rupture life of the base composition 16 pct Cr-18 pct Mn-2 pct Mo-½ pct N are shown on a Larson-Miller plot.

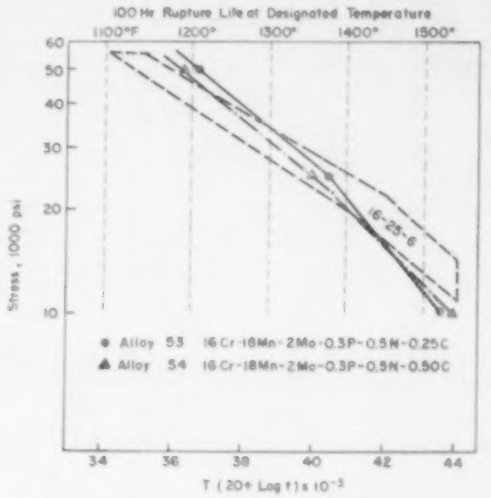

Fig. 2—Creep-rupture strength of modified base composition, 0.25 pct C phosphorus steel, is shown.

stress for rupture is plotted against a parameter involving temperature and time to rupture. For convenience, dotted vertical lines defining the 100 hr rupture life at the indicated temperatures are shown. Representative values for the normal scatter of wrought creep-rupture properties of commercial alloy Timken 16-25-6 taken from several sources in the published literature are shown as a band superimposed on the Larson-Miller plot. The creep-rupture parameters for the as-cast base composition, alloy 49 C, are seen to lie well within the wrought Timken 16-25-6 band at parameter values corresponding to short time rupture tests up to 1400°F. The test results of the as-cast material are almost identical with those obtained on wrought material of the same composition.

To gain the potent hot strengthening characteristics of phosphorus and at the same time to offset its ferrite-forming tendency, a series of heats containing both carbon (a strong austenitic former) and phosphorus were made. In these alloys carbon performs the dual function of hot strengthening and stabilizing the austenite matrix. The excellent creep-rupture strength of the low carbon (0.25 pct) phosphorus steel, alloy 53, is shown in Fig. 2. Further additions of carbon (0.50 pct) decreases the hot strength of this steel, alloy 54, although its properties are still nearly equivalent to those of wrought Timken 16-25-6. Alloys 53 and 54 were tested in the solution quenched and aged condition (100 hr at 1250°F).

Several cast austenitic Cr-Mn-Mo-N steels have been investigated for possible service in the 1200° to 1400°F temperature range. A base composition of 16 pct Cr-18 pct Mn-2 pct Mo-½ pct N proved to have creep-rupture strengths equivalent to wrought Timken 16-25-6 in the temperature interval cited previously. Several modified alloys containing small amounts of carbon and phosphorus exhibited heat-resistant properties superior to those of the base composition. These high nitrogen steels appear to have promise in view of the need for a cast iron-base austenitic material of low strategic index capable of service above the usual operating temperatures of the best ferritics and below those of the so-called superalloys.

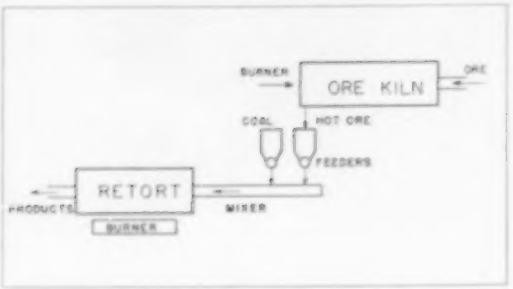

Fig. 1—Flow diagram of the Orcarb process for agglomerating fine ores with low temperature coke shows the pilot operation as it is now constituted.

Agglomerating Fine Sized Ores with Low Temperature Coke

by C. E. Lesher

Two processes for agglomerating fine sized ores with low temperature coke are described. One process (Orcarb) agglomerates ores with limited amounts of carbon; the other (ore-carbon pellets) pelletizes fine sized ores, using low temperature cokes as the binder. Data are presented on the products obtained when taconite, magnetite, and hematite concentrates and several titanium oxide ores were used.

REDUCTION of finely divided oxide ores has long been a major metallurgical problem. Various methods of agglomerating, notably briquetting, sintering, nodulizing, and pelletizing, have been developed and are in industrial use. Carbon is the usual reducing agent for oxide ores and attempts have been made to agglomerate fine ores with coking coal in order to get the metallurgical advantages of intimate contact and increased particle sizes suitable for subsequent smelting or reduction.[1-4]

For the past three years, research has been in progress on two processes for combining fine sized ores with reactive carbon in the form of low temperature coke. By one process, designated *Orcarb*, the ore is coated with coke carbon in amounts limited to that required for reduction and the result is an appreciable but limited increase in product particle size over that of the original ore. The second process, called *ore-carbon pellets*, produces pellets of fine ore bound by low temperature coke.

The first objective was to develop a process for combining an oxide ore with only enough carbon for its reduction, the amount being that derived from the simple reduction equations. The carbon (that amount theoretically required for the reduction of the oxides in an agglomerate with ore), calculated to CO, ranges from 10 pct for zinc calcines or phosphate ores to 21 pct for rutile and 26 pct for silica. The carbon required for reducing iron oxides falls between the extremes—10 and 26 pct. When the carbon in the agglomerate is low temperature coke, reduction by hydrogen may be expected to take place, thus modifying the calculated carbon requirement. On the other hand, in practice, an excess of reducing agent over that required in theory is normally used.

Screen sizes of three iron ores on which the greater part of the experimental work was performed are given in Table I. Most of the testing was done with Freeport (Renton Mine) coal, but Pittsburgh and Elkhorn bed coals were also used.

Table I. Screen Sizes of Certain Iron Ore Concentrates

Screen Size, Tyler Mesh	Taconite, 66.5 Pct Fe		Hematite, 69.6 Pct Fe		Magnetite, 63.8 Pct Fe	
	Pct	Cumulative Pct	Pct	Cumulative Pct	Pct	Cumulative Pct
On 4	—	—	1.5	1.5	—	—
4x8	—	—	0.4	1.9	—	—
8x14	0.05	0.05	0.4	2.3	0.8	0.8
14x28	0.10	0.15	0.8	3.1	19.8	20.6
28x48	0.23	0.38	5.8	8.9	32.9	53.5
48x100	2.22	2.60	24.6	33.5	24.8	78.3
100x200	23.40	26.00	23.0	56.5	11.7	90.0
−200	74.00		43.5		10.0	
	100.00		100.0		100.0	

Orcarb Process

The first objective was to agglomerate the ore with sufficient low temperature coke to provide carbon for reduction, i.e., in the range from 10 to 25 pct. This was accomplished by preheating the ore, mixing with pulverized coking coal, and completing the coal carbonization in a plain steel rotating retort.

The pilot retort, as it is now constituted, is shown by diagram in Fig. 1 and photograph, Fig. 2. Ore is fed into the upper kiln by a screw; it is heated to 900° to 1100°F by direct flame and it is then discharged into an insulating hopper from which it goes

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at a predetermined rate into a mixing conveyer. Pulverized coal also goes to the mixing conveyer at a measured rate from another hopper. The mixture is continuously charged into the lower externally heated retort where the operation is completed and from which the solid product, Orcarb, as well as tar vapor and gas, is continuously withdrawn. The process depends upon bringing the fine sized heated ore particles into contact with the coking coal in such a temperature relationship that the ore is coated with low temperature coke before the denser ore and lighter weight coal segregate. At first, softened bituminous material coats the ore; then the finest sized coated particles will be bonded into larger aggregates if the bituminous material is more than sufficient to coat and impregnate the ore particles.

Uniformity of product, in both size and analysis, depends upon charging the coal into coke while it is in contact with the ore. To accomplish this, the average temperature of the raw coal and ore in the mixer is maintained just below the softening point of the coal; the two materials are well mixed and charged into the revolving retort where they are immediately further heated to complete the carbonization of the coal.

Table II. Analyses of Orcarb by Size*

Screen Size Mesh		Wt Pct		Ignition Loss, Pct	
On	Through	A	B	A	B
6	—	4.2	5.0	14.7	7.3
10	6	8.3	8.0	18.5	17.1
14	10	8.0	7.7	18.2	18.0
20	14	11.5	13.5	18.4	17.5
60	20	42.1	45.0	14.7	14.1
100	60	6.7	6.7	10.7	10.5
—	100	19.2	14.1	4.8	4.0
		100.0	100.0		
Weighted average, total		—	—	13.5	13.1
+60 mesh		—	—	16.1	15.0

* Total sample of Orcarb weight 3600 lb.

In this process, the percentage of ore is decreased as the percentage of carbon is increased. This relationship is shown in Fig. 3 where the iron percentages are plotted against the carbon percentages for a series of tests on two different iron ores. The percentages of carbon were determined by combustion, using the procedure for ultimate analysis of coal or coke.

Size distribution and relative analyses of Orcarb made from taconite concentrates are shown in Table II. These figures represent the totals obtained from the production of 3600 lb +60 mesh Orcarb from taconite concentrates. The product that passed the 60 mesh screen was recycled. Each 100 lb charge that was processed contained approximately 17 lb coal, 63 lb ore, and 20 lb −60 mesh recycle. Total carbon in the product was 13.5 pct. Carbon in the +60 mesh product was 15.5 pct. The data in col. *A*, Table II, covers two-thirds of the total production and that in *B*, the final third. The figures designated *ignition loss* are 100 pct less ash for each sample. Ignition loss represents an approximation of the carbon and volatile matter in the product.

Three series of tests, in which the ratio of coal to ore charged was varied, are recorded in Table III. As indicated by the data in this table, the granular product of the Orcarb process is largely within the 6x60 mesh range and the amount of carbon in the Orcarb may be varied from 10 to 25 pct as desired. Of the series 1 (Table III) Orcarb, which contained over 21 pct C, 65 wt pct fell in the +20 mesh size, and two-thirds of 65 wt pct product was +6 mesh.

Table III. Screen Sizes and Analyses of Orcarb*

Size Mesh	Series 1	Series 2	Series 3
	Total Carbon, Pct		
+20	25.0	21.0	19.4
60x20	20.6	16.3	15.8
−60	8.0	6.5	4.0
Average	21.7	14.3	11.3
	Total Iron,† Pct		
+20	45.7	47.9	48.1
60x20	49.5	51.7	53.2
−60	56.0	58.0	60.0
Average	48.0	52.6	55.2
	Wt, Pct		
+20	65.0	31.9	19.5
60x20	16.5	34.6	36.5
−60	18.5	33.5	44.0
	100.0	100.0	100.0

* Orcarb was produced from taconite concentrates and high volatile coking coal.
† Analyses of a number of samples from these tests gave 14.0 to 16.0 pct of the total iron as FeO.

Less than 2 pct of the total weight of the Orcarb in series 2 and 3 was +6 mesh.

The percentage of carbon in the product is limited, by certain metallurgical processes, to that required for reduction; these processes introduce process heat from sources other than by the combustion of carbon in the charge. Some examples in which the process heat applied is independent of that available in the carbon charge are the reduction of zinc oxide in retorts, iron ore in the electric arc furnace, and titanium oxide in the chlorination process. Orcarb will meet the carbon requirements and, for some uses, the size requirements. It will be suitable for use in a fluid reactor, but because of the small size of the product, it will not be suitable for a fixed bed reactor. Orcarb has been found to be an efficient material for reducing iron in the electric open arc furnace.

Ore-Carbon Pellets

The next step in the study of agglomerating fine sized ores with low temperature coke was to develop a process for producing large particle sized aggregates for use in the fixed bed reactor. Thus, the objective became the control of product particle size rather than limited carbon content as discussed previously. Assuming that more coal would automati-

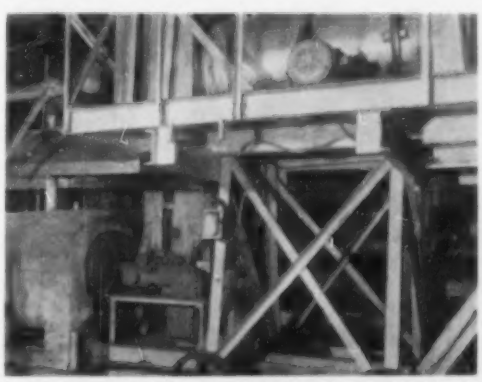

Fig. 2—Orcarb pilot plant is used for agglomerating the ore with low temperature coke. On the upper right is the ore preheating kiln; in the center left are the insulated hot ore and coal hoppers from which the coal and ore are charged to the mixing conveyer; and on the left is the charge end of the externally heated retort. The kiln and hoppers are separately mounted on carriages and can be withdrawn so that the interior of the retort can be inspected.

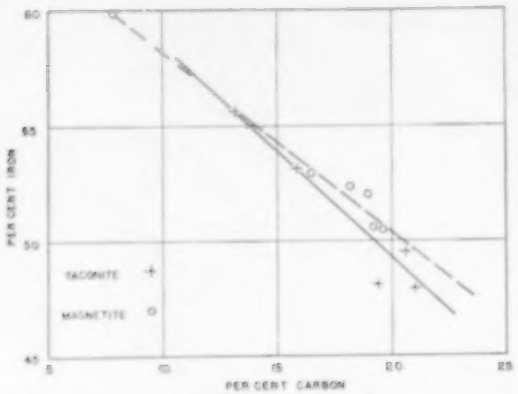

Fig. 3—In the Orcarb process, the percentage of ore is decreased as the percentage of carbon is increased. The graph shows this relationship for two ores, taconite and magnetite concentrates, and coking coal.

cally create larger product, tests were made with the same ores, coal, and procedure used for manufacturing Orcarb. The results were unsatisfactory. No product control resulted, and ore and coal segregated in the retort. The product consisted of irregular masses of coke with low ore pick-up and of much fine and broken material with a high iron content. Cokey crusts built up on the retort walls and on the end of the feed screw.

After conducting tests for several months with different ores and coals, it was decided to try pelletizing these raw materials in the way developed for processing ores with water binder.

Fine sized ores are currently being rolled into pellets with water for a binder. These pellets or *mud balls* are dried, baked, and utilized in subsequent processing. The operation is simple: ore is wetted with water, rolled at atmospheric temperature in a plain steel horizontal cylinder, roughly sized, and dried sufficiently for handling. The variables in the process are the percentage of water binder, diameter and length of the kiln or retort, and the speed of rotation. It appears that the most important variable is the percentage of water used; this may vary with the different charge materials.

Fig. 4 — Photograph of the interior of the ore-carbon pelletizing retort was taken of the operation under load. The retort was halted in process and the load allowed to cool. The unconsolidated charge mixture of coking coal and iron ore concentrates can be seen in the foreground. The mixture is changing to pellets in the center and formed pellets can be seen in the background at the discharge end. The curved pipe in the upper right contains a thermocouple.

The peripheral speed of the pelletizing drum must be such that it carries the wet charge up but not over the drum. Wet pellets are fragile. Different devices and procedures are being used for drying and subsequent hardening, baking, or calcining.

In the process being discussed, plastic coal is used as the binder for fine ore pellets and the pellets are rolled and hardened by the coal in one operation. The one operation, of binding, rolling, and hardening, is accomplished by bringing fine sized ore and coking coal in intimate mixture to the pelletizing drum or retort at a temperature approaching that of the softening point of the coal; the temperature in the retort is increased in order to make the coal plastic as the mixture is rolled. The operation is continuous and the pellets, as they are formed, travel from feed to discharge end of the retort and are removed as solid pellets. Heat to complete the coal softening and to carry it through the plastic condition to solid form is put into the charge through the steel shell of the retort. Tar vapor and gas are withdrawn, of course, as they are produced.

The ore is heated in a direct fired rotary kiln to between 800° and 900°F and is delivered continuously to an insulated mixing conveyer. The coal is fed continuously into the same mixer. The materials are allowed to remain in the mixing conveyer only about 1 min. As the mixture leaves the mixing conveyer, it has an average temperature below the softening temperature of the coal and is fed into one end of the revolving pelletizing retort. This retort is heated by external burners and the temperature of the charge of coal and ore inside is raised at such a rate that the coal becomes plastic as it is being rolled with the ore. Pellets are formed and travel along the retort to the discharge end. Sufficient heat is applied so that the temperature of the product inside the retort and at the discharge end is maintained at 900° to 950°F. The pelletizing process has been accomplished with ores largely finer than 20 mesh and with coking coals having the plastic qualities of either the Pittsburgh or the Freeport beds.

Fig. 4 shows the interior of the pelletizing retort where the material is being changed from loose charge (bottom) to pellets (top). To obtain this picture, the retort was started in normal fashion: ore was charged through the preheating kiln at the rate of 2.5 lb per min and then mixed with the coal which was fed at the rate of 1.2 lb per min. After 45 min of normal operation, the process was sud-

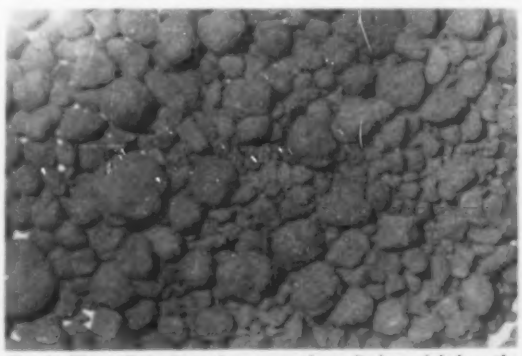

Fig. 5—The pellets shown here were those discharged before the operation was halted for the photograph in Fig. 4. The rough surfaces have been polished and many of the larger pellets have been broken during handling through the discharge screw. A large part of the —¼ in. material in the product can be accounted for by abrasion.

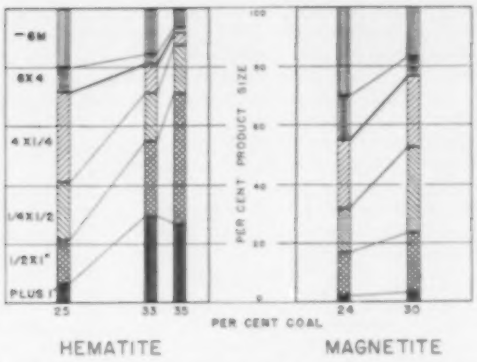

Fig. 6—The diagram shows the product sizes of ore-carbon pellets produced from fine sized hematite and magnetite ores and increasing percentages of coal. The hematite concentrate was the largest ore in order of pellet size and the magnetite was next.

denly stopped, the burners shut off, and the unit sealed and allowed to cool over night. The retort in Fig. 4 was rotating counterclockwise. The loose unconsolidated charge is seen at the bottom of the photograph and the finished pellets, which are ready to be carried up and into the discharge screw, are seen at the top. The material in the center, between the loose charge and the finished pellets, was between 2 and 3 in. thick and solid when cold. When the solid mass was examined, it was seen that, within 12 to 18 in. from the charge end, pellets were being formed in what was evidently a matrix of plastic material. In the center of the 4 ft long retort, pellets predominated and beyond that point all the material was in pellet form—at first sufficiently plastic to solidify to mass when cold, but at the discharge end comprised of solid separate pellets.

Before the retort was stopped for the photograph shown in Fig. 4, the screen analysis of the product already discharged (Fig. 5) was: 27.4 pct 1x1½ in., 43.7 pct ½x1 in., 16.3 pct ¼x¼ in., 4.4 pct 4 mesh by ¼ in., 0.7 pct 6x4 mesh, and 7.5 pct −6 mesh, totaling 100 pct.

Pellet Product

The pellet product consists of generally rounded particles, the average size of which depends upon the ore particle size and the relative amount of coal in the charge. The size distribution of pellets made from four different ores pelletized with increasing percentages of coal are shown in Figs. 6 and 7. The effect of ore particle size on product size as increasing percentages of coal are used is brought out more clearly in Fig. 8. In this diagram, the weight percent of +¼ in. pellet product produced from each ore is related to the amount of coal charged with the ore. The increase in size is more or less directly proportional to the amount of coal used; the coarsest ore shows 20 to 25 pct more +¼ in. product than the finest. The weighted average size of the pellet product over 6 mesh varies from about 0.275 to 0.5 in. Except for the coarsest ore tested, the weighted average sizes of +6 mesh fall in the range of 0.3 to 0.4 in.

Undersize pellets may be recycled. In the tests that have been made on the recycled material, it has been found that, although a −6 mesh material is much larger than the ore, there is no corresponding gain in product particle size. No doubt this is due to the porosity of the recycled material, since it re-

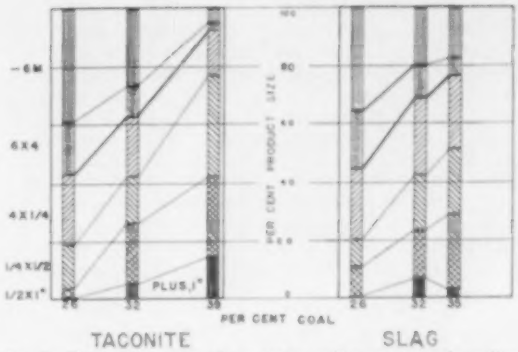

Fig. 7—The product sizes of ore-carbon pellets produced from fine sized taconite ore and slag and increasing percentages of coal are shown in the diagram. Of the four materials shown in Figs. 6 and 7, the taconite concentrates were the finest in particle size. The ilmenite furnace slags were larger than the taconite but smaller than the hematite (largest) and magnetite (second in size).

quires more binder than denser and finer sized ore. This condition can be corrected by pulverizing the material to be recycled. The bulk density of the pellet product made from −6 mesh recycle was 46 lb per cu ft, as compared with 66 lb for product made from ore, bulk density of which was 117 lb.

Analyses

Analysis of the low temperature coke in the agglomerated products has been calculated from the analyses of agglomerates made from an oxide ore (rutile) that is neither oxidized nor reduced during processing. The results presented in Table IV for two tests were derived by first calculating to moisture-free and ash-free bases and then adjusting to the basis of 6 pct ash coke.

Physical Properties of Pellets

It would be desirable, of course, to compare the physical strength of the pellets with other sized material of like nature, in the way in which cokes

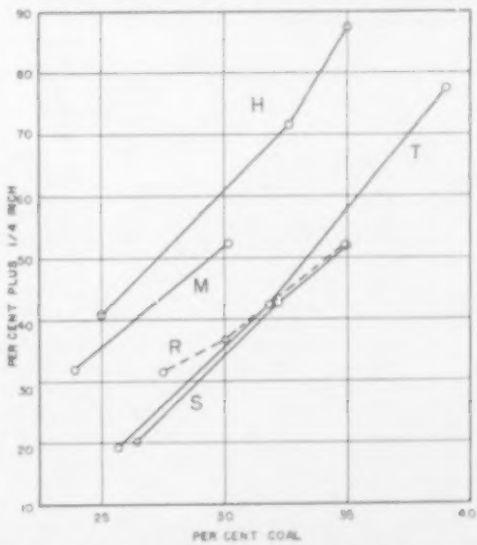

Fig. 8—In the diagram, the four ores of Figs. 6 and 7 are compared with respect to pellet size, with the addition of data from tests with rutile. *H* stands for hematite concentrates; *M*, magnetite concentrates; *R*, rutile; *S*, ilmenite furnace slags; and *T*, taconite concentrates.

are compared by shatter and tumbler tests and fuel briquets by crushing, accelerated weathering, and tumbler tests. Taconite pellets, after drying and baking at 2000°F (more or less) in vertical shaft retorts, are compared for resistance to abrasion and breakage by a tumbler test in the device used for stability testing of furnace coke.

Table IV. Analysis of Low Temperature Coke in the Agglomerated Products

	Test No. 100		Test No. 130	
	Moisture and Ash-Free	Dry	Moisture and Ash-Free	Dry
Volatile matter	17.5	16.5	23.0	21.6
Fixed carbon	82.5	77.5	77.0	72.4
Ash	—	6.0	—	6.0

Actually these ore-carbon pellets are tumbler tested during the manufacturing process. The pellets, while plastic during formation, are rounded and compacted by the centrifugal force imposed in the rotating drum. After solidification, they are as effectively tumbled and tossed as they would be in any other device. Product of less than ¼ in. is believed to be largely the result of this tumbling.

Temperature Susceptibility

Some concern has been expressed over the belief that agglomerates of ore and low temperature coke are highly susceptible to shock when exposed to the temperatures at which reducing reactions take place; the author, however, can find no evidence to support this assumption. On the contrary, there is evidence that this kind of agglomerate is not subject to disintegration, explosion, or shattering when heated to high temperatures, either gradually or suddenly. After exposure to the effects of weather for several years, 2 and 3 in. pieces of Ore-Disco were dropped into a molten slag bath in an electric furnace and withdrawn after about 15 min. They did not shatter, crumble, or disintegrate. No breakdown in size resulted after samples of ore-carbon pellets were heated to 1700°F in closed cast iron pots. The only effect of such treatment seemed to be hardening of the pellets and, of course, reduction in the volatile matter content.

Tests have been made and are continuing on the use of these pellets, manufactured from rutile and ilmenite ores, in chlorination fixed bed processes. The information that is available shows that the pellets are well formed, strong, and readily chlorinated.

Summary

Ore concentrates are pelletized, using low temperature coke as a binder. Fine sized preheated ore and pulverized coking coal are rolled to pellet form and hardened continuously in a rotating externally heated retort. The carbon in the product may be varied from 25 to more than 40 pct. With a given ore, the pellet product size increases as the amount of carbon is increased. These pellets are adapted for oxide reduction in fixed bed reactors and buried arc electric furnaces.

By another process, a product called Orcarb is made from ore concentrates or calcines; in this process the ore particles are coated with a predetermined and limited amount of carbon (in the range of 10 to 25 pct), and the product used is largely between 10 and 60 mesh. Orcarb is suitable for use in fluid bed reactors, open arc electric furnaces, or other operations in which the process heat is externally applied.

Discussion of this paper, if any, will appear in JOURNAL OF METALS, November 1956, and in AIME Metals Branch *Transactions*, Vol. **206**, 1956.

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[2] United States Patent No. 2,029,309.
[3] C. E. Lesher: Low Temperature Coke as a Reactive Carbon. *Trans.* AIME (1950) **187**, pp. 805-810; MINING ENGINEERING (July 1950).

Preparation of High Purity $ZrCl_4$ from Alkali Chlorozirconates

by Robert V. Horrigan

High purity $ZrCl_4$ (25 to 200 ppm of metallic impurities) can be readily prepared in simple apparatus by thermal decomposition of alkali chlorozirconate fused salts at 500° to 600°C and at atmospheric pressure. Suggested phase diagrams for the systems NaCl-$ZrCl_4$ and KCl-$ZrCl_4$ are presented.

ALKALI chlorozirconate compositions—anhydrous fused mixtures of $ZrCl_4$, NaCl, and KCl—are manufactured commercially and are used by the magnesium industry to introduce zirconium into magnesium alloys. Zirconium refines the grain size and improves the physical properties of many important magnesium alloys. In the course of working with chlorozirconate compositions of high $ZrCl_4$ content, it was noticed that these salts decomposed progressively with increasing temperature to yield considerable amounts of a $ZrCl_4$ sublimate of high purity together with a solid chlorozirconate residue. Further research led to: 1—improvements in the

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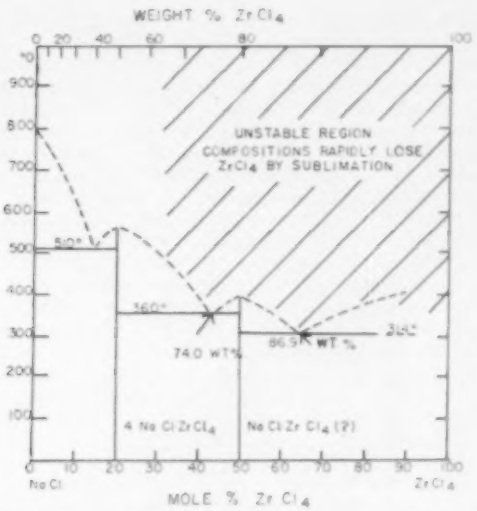

Fig. 1—Suggested phase diagram is presented for the system NaCl-Zr_4 and was constructed from the author's data and those of Howell et al.[1] and Kroll et al.[2]

yield and purity of the $ZrCl_4$ product, 2—determination of the optimum composition of the salts, and 3—refinements in apparatus. It was also found that the residue salt could be remelted with additional crude $ZrCl_4$, thus making possible a cyclic process for purification of impure $ZrCl_4$.

Phase Diagrams

The probable phase diagram for the system NaCl-$ZrCl_4$ is presented in Fig. 1. This diagram is based partly on data developed by the author but principally on the data of Howell et al.[1] and the data of Kroll et al.[2] The earlier work of Belozerski and Kucherenko,[3] which placed the three eutectics at 390°, 220°, and 162°C, is believed to be in error. The melting point for pure $ZrCl_4$ is given as 437°C, the calculated melting point for this compound. Actually, $ZrCl_4$ sublimes at 331°C at 1 atm pressure. The diagram shows that the lowest eutectic point, 314°C, occurs at 62.4 mol pct (86.9 wt pct) $ZrCl_4$.

Since $ZrCl_4$ sublimes at 331°C, it is desirable in the manufacture of chlorozirconate compositions of high (70 to 90 wt pct) $ZrCl_4$ content to use the proportions of alkali metal chloride and $ZrCl_4$ which will melt at the lowest possible temperature. Early research at the National Lead Co. laboratory was centered around a 50-50 by weight mixture of NaCl and KCl, with which varying amounts of $ZrCl_4$ were melted. The lowest eutectic in this system, a composition containing 75.0 wt pct $ZrCl_4$, was found to melt at 240°C. From 75 down to 65 wt pct $ZrCl_4$, the melting point did not exceed about 260°C, but at 50 wt pct $ZrCl_4$ (the approximate composition of one of the chlorozirconate products which has been used commercially), the melting point jumps to 580°C; this sudden rise in melting point indicates a compound similar to 4NaCl-$ZrCl_4$.

A tentative phase diagram for the system KCl-$ZrCl_4$ is presented in Fig. 2. The compound 4KCl-$ZrCl_4$ (44.1 wt pct $ZrCl_4$), which is approximately the composition of another commercial chlorozirconate product, melts at about 620°C. In this system, the lowest eutectic composition melts at about 215°C and occurs at 88.0 wt pct $ZrCl_4$. The existence of the compound KCl-$ZrCl_4$ is suggested by the data which indicate the presence of three eutectics. The exact compositions of the first two eutectics have not yet been determined.

Table I. Typical Yields of High Purity $ZrCl_4$ by Thermal Decomposition of 88 pct $ZrCl_4$-12 pct KCl Fused Salt

Decomposition Temperature, °C	Time at Temperature, Hr	Yield of $ZrCl_4$, Parts per 100 Parts Fused Salt
400	1	37.5
450	2.5	61.1
500	1	64.0
600	1	62.5

Thermal Decomposition of Alkali Chlorozirconates

Alkali chlorozirconate compositions in the range of about 70 to 90 wt pct $ZrCl_4$ decompose readily at temperatures above their melting points to yield a high purity $ZrCl_4$ sublimate and a solid residue containing less $ZrCl_4$ than the original composition. In particular, the eutectic fused salt containing 88 wt pct $ZrCl_4$ and 12 wt pct KCl can be easily and efficiently prepared because of its relatively low melting point (215°C) with respect to the sublimation temperature of $ZrCl_4$ (331°C). In addition, good yields of high purity $ZrCl_4$ are recovered by heating this fused salt composition for comparatively short times to 500°C or above. Table I gives typical $ZrCl_4$ yields for decomposition of this fused salt composition at various temperatures. The residue chlorozirconate composition, depending on the decomposition temperature and decomposition time employed, was found to contain about 65 to 70 wt pct $ZrCl_4$. It was found feasible to reuse this residue by remelting it with the proper amount of crude $ZrCl_4$ to return the composition of the molten salt to about 88 wt pct $ZrCl_4$ and then to proceed with the preparation of more high purity $ZrCl_4$. It is estimated that such recycling of the residue chlorozirconate could be continued for about 50 to 100 cycles (depending on the quality of the $ZrCl_4$ to be purified) before the trapped impurities would be raised to the point where the purity of the $ZrCl_4$ sublimate would begin to be affected.

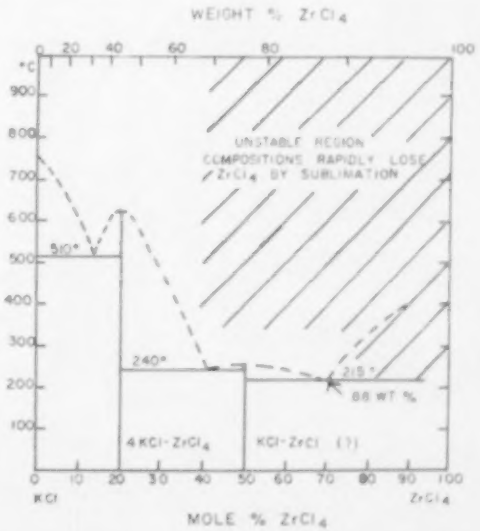

Fig. 2—Suggested phase diagram is presented for the system KCl-$ZrCl_4$.

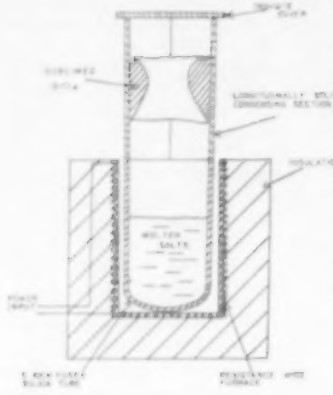

Fig. 3—Sectional view shows the apparatus used for the preparation and decomposition of the alkali chlorozirconate salts. This equipment also provided a means of collecting the high purity $ZrCl_4$ sublimate.

The purity of the $ZrCl_4$ sublimate is illustrated by typical spectrographic analyses shown in Table II. Typical analyses of crude $ZrCl_4$ (produced by chlorination of zirconium cyanonitride) and $ZrCl_4$ sublimed under hydrogen gas are included for purposes of comparison. The amount of oxygen in the $ZrCl_4$ sublimate was roughly estimated as follows: a quantity of the 12 pct KCl-88 pct $ZrCl_4$ salt was placed in the bottom of a long dry Pyrex tube sealed at one end and equipped with a bunsen valve at the open end. The salt was decomposed in the usual way, the $ZrCl_4$ sublimate collecting in a ring some distance above the heated zone. The sublimate was then carefully and slowly resublimed. Assuming that the residue remaining after this resublimation was ZrO_2, the amount of oxygen in the sublimate when formed by thermal decomposition of the 12 pct KCl-88 pct $ZrCl_4$ salt was estimated to be no more than 30 ppm O in zirconium by weight.

Apparatus for Thermal Decomposition of Alkali Chlorozirconates

Fig. 3 depicts a piece of apparatus which proved convenient for the preparation and decomposition of the alkali chlorozirconate salts and also provided means of collecting the high purity $ZrCl_4$ sublimate. A fused silica tube, 5 in. diam and closed at the bottom, is placed in a resistance wire-wound furnace capable of raising the temperature of the tube contents to 700°C. The top of the silica tube is level with the top of the furnace liner. The mixed alkali chloride and $ZrCl_4$ salts are placed in the tube and melted at the desired temperature. When all the tube contents are molten, a 5 in. diam cylinder of fused silica which has been carefully cut into halves along the length of the cylinder is placed on top of the tube containing the molten salt. The two halves of the cylinder fit closely together and are held in place by tying with asbestos cord. A close-fitting transite cover is placed on top of the split cylinder. The molten chlorozirconate salt is then decomposed by raising the temperature of the bath to about 500°C for 1 to 2 hr, during which time the apparatus is under a positive pressure of $ZrCl_4$ vapor. The high purity $ZrCl_4$ product in the form of a large ring collects as a solid crystalline mass on the inside walls of the air-cooled cylindrical condenser. Removal of the $ZrCl_4$ ring is facilitated by the split-wall construction of the condenser. A slight amount of surface contamination of the product by atmospheric moisture does take place during collection of the product, but moisture does not penetrate into the crystalline mass.

Table II. Typical Spectrographic Analyses of Crude and Hydrogen-Sublimed $ZrCl_4$ Compared with $ZrCl_4$ Prepared by Thermal Decomposition of 88 Pct $ZrCl_4$-12 Pct KCl Fused Salt*

	Typical Crudes†			Hydrogen Sublimed†	Typical Fused Salt Sublimates†			
Element	1	2	3	4	5	6	7	8
B	100	—	—	100	—	—	—	—
Si	1200	200	120	1200	25	30	3	2
Sb	—	20	—	—	—	—	—	—
Fe	800	2000	3000	20	5	10	20	5
Cr	—	15	—	—	—	—	—	—
Mn	1	10	3	—	—	—	—	—
Pb	—	12	—	—	—	—	—	—
Mg	100	50	8	50	1	10	1	2
Sn	—	2	—	—	—	—	—	—
Ti	40	300	8	20	2	—	3	3
Al	70	400	35	70	15	10	7	7
Ca	2	—	10	2	—	—	—	—
Cu	100	20	20	50	20	20	2	2
Zn	—	50	—	—	—	—	—	—
Ba	d	—	5	6	5	—	—	—
Na	100	—	100	10	100	—	5	5
K	—	—	—	—	20	—	—	—
Ni	—	30	—	—	—	—	—	—
Total	2519	3129	3309	1528	190	80	41	26

* Values are in parts per million parts of zirconium (including hafnium—commercial $ZrCl_4$ contains approximately 2 pct $HfCl_4$; except for nuclear applications, hafnium is not considered to be an impurity) and are approximations according to usual spectrographic practice.

† A dash signifies that the element was not detected.

Proposed Mechanism for the Observed Purification of $ZrCl_4$

Crude $ZrCl_4$ is usually contaminated with both nonvolatile and volatile impurities. Except for unavoidable carry-over of fines, most of the nonvolatile impurities can be eliminated by sublimation of the crude $ZrCl_4$. However, volatile contaminants such as $FeCl_3$, $AlCl_3$, $SiCl_4$, $TiCl_4$, and others, generally are not completely removed from the $ZrCl_4$ during sublimation. It is probable that, during the reaction of the alkali chloride and the $ZrCl_4$ to form the alkali chlorozirconate salt, most of the $SiCl_4$ and $TiCl_4$ impurities distill off while the $FeCl_3$, $AlCl_3$, and other metal chloride impurities form stable alkali chloride double salts. The presence of any moisture will, of course, make these metal chlorides much less volatile. During thermal decomposition of the chlorozirconate salt, the molten bath serves the twofold purpose of physically trapping the nonvolatile matter and chemically binding those metal chloride impurities which can form alkali chloride double salts. This is the mechanism, it is believed, that results in the observed high purity of the $ZrCl_4$ sublimate.

Acknowledgment

Acknowledgment is made of the contribution of Owen F. Sprague, formerly of this laboratory, in connection with the initial work on this development.

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475°C Embrittlement of Chromized Coatings On Low Carbon Steel

by W. L. Chu

It was observed that chromized coatings on low carbon steel could be embrittled by cooling slowly through the 475°C range. A higher chromium coating was more susceptible to embrittlement than a lower chromium coating. Coating structure and composition could be correlated with the chromizing process and prior carbon content of the steel.

IT was recently observed that during the fabrication of rectangular tubings made with chromized thin gage 1010 steel, a high percentage of these parts were fractured readily in a brittle manner under the light impact-forming load employed. Ensuing investigation indicated the cause for such brittle failures to be the so-called 475°C embrittlement of the chromized coatings. The experimental work and results are briefly reported.

Rectangular tube sections of normalized AISI C1010 steel, which had a cross-section of 0.5x1.5 in. and a wall thickness of 0.030 in., were chromized to yield coatings having several ranges of thickness, varying from 0.0005 to 0.002 in. Essentially, the chromizing process is similar to that described by Samuel and Lockington,[1] which consists of heating the parts in a sealed container at about 955°C for 4 hr in intimate contact with a chromizing mixture*

* The mixture nominally contains 65 pct ferrochrome (50 to 70 pct Cr), 0.1 pct I, 0.25 pct ammonium iodide, and the balance unvitrified kaolin.

in a reducing atmosphere. The parts are slowly cooled to near room temperature in the same atmosphere before discharge.

A simple hammer test was adopted for testing the brittleness of the prepared samples. This method involved striking light hammer blows at several areas of the sample, the force of each blow being sufficient to cause complete flattening of the area tested. Out of 45 samples prepared for the study and tested, 13 cracked readily at each test area and the rest were flattened in a ductile manner without cracking. The former were classified as brittle samples and the latter as tough. Thus, two groups of contrasting physical behavior were available for study. A representative sample of each type is shown in Fig. 1 with an undeformed sample for comparison.

Two other tests for brittleness were also attempted. The first involved slowly flattening the

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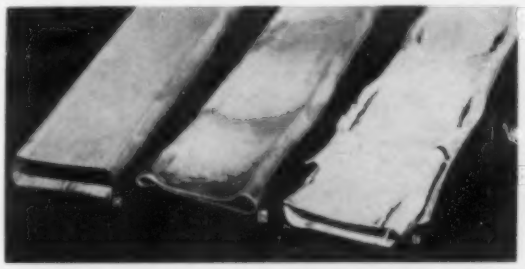


Fig. 1—Two groups of chromized samples of contrasting physical behavior were available for study; an undeformed sample is shown for comparison. a—Chromized sample before hammer test. b—A tough chromized sample after testing. c—A brittle chromized sample after testing.

samples in a hydraulic press, and the other employed a simple beam bending test of the flat sides of the tubing walls. Neither, however, was able to differentiate the relative brittleness of samples known to include both the brittle and tough types, since the impact-brittle samples were found to deform readily in a manner similar to the tough ones.

Both the tough and brittle samples were studied by reflection microscopy. Observation showed that the microstructures of the steel cores were similar in content and distribution of inclusions as well as in the ferritic grain size. Results of Filar micrometer measurements showed that coating thicknesses† of

† Coating thickness refers to the distance from the surface to the interface where the chromium content is approximately 13 pct.

the brittle samples averaged 0.0007 in., whereas those of the tough samples averaged 0.0016 in. The micrographs of Fig. 2 illustrate the structural differences between coatings of both types of samples.

By dissolving the steel cores in nitric acid, coatings of both the brittle and tough samples were isolated. Simple bending tests showed that coatings of the brittle samples cracked readily at about 90° bend, whereas those of the tough samples could be bent nearly 180° without cracking. The coatings as well as the steel cores were analyzed for carbon and

chromium contents. The light etching constituents in some of the brittle coatings were extracted and also analyzed for the two elements. The results are listed in Table I.

It was suspected that the impact brittleness of the samples was associated with the brittleness of the coatings and quite probably with the 475°C embrittlement of the coatings during the chromizing operation. Thus, heat treatments were run which were believed to give information of an enlightening nature. Sections of the brittle samples were heat-treated at 700°C for 30 min in a dry hydrogen atmosphere and were cooled at an average rate of 25°C per min through the 475°C range. Subsequent hammer test showed that the samples were considerably toughened and could be flattened without cracking. By reheating to 700°C for 30 min and cooling through the 475°C range at an average rate of 0.4°C per min, the samples were found to regain their original impact brittleness. Examination by microscope of the samples after each treatment revealed no structural changes at magnifications up to X2000. When the tough samples were subjected to the same slow cooling treatment, their impact-forming behavior was altogether unaffected and remained tough throughout the hammer test.

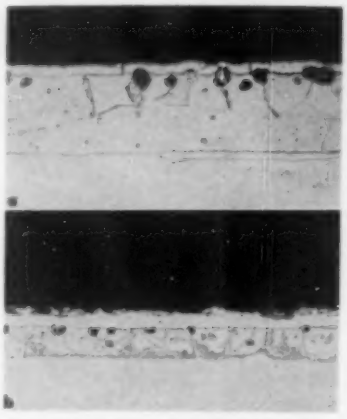
Table I. Analyses of Brittle and Tough Chromized Samples

Sample Group	Impact Characteristic	Portion Analyzed	Pct C	Pct Cr	Pct Fe
A	Tough	Coating and core	0.043	—	—
B	Brittle	Coating and core	0.070	—	—
C	Tough	Coating	0.90	24.0	Balance
		core	0.018	—	
D	Brittle	Coating	1.80	32.4	Balance
		core	0.047	—	
E	Brittle	Extracted residues	4.90	71.0	Balance

To determine the effect of further diffusion (and hence lowering of the average chromium content and alteration of microstructure) in the coatings of brittle samples on their susceptibility to 475°C embrittlement, sections of these samples were heat treated in dry hydrogen according to the following procedures: 30, 60, 120 min at 850°C, 30, 60, 120 min at 915°C, 30 and 60 min at 1000°C, and 30 min at 1150°C. Water quench followed each treatment.

Microscopic examination showed that heat treatment at 850°, 915°, and 1000°C for as long as 120 min did not result in any change in either microstructure or thickness of the brittle coatings. However, treatment at 1150°C increased the coating thickness by an average of 85 pct, caused extensive changes in the coating structure, the most notable of which being the disappearance of the outer layer, the precipitates, and the intergranular constituent, and accompanied growth of the columnar grains; see Fig. 3. The 1150°C treated samples were later reheated to 700°C for 30 min and slowly cooled through the 475°C range. Hammer tests showed that the samples were tough.

The foregoing results indicate that the impact brittleness of certain chromized low carbon steel parts was due to embrittlement of the chromized coatings when the parts were slowly cooled through the 475°C range, most probably at the end of the chromizing process. It is surmised that, under impact loading and the mechanical restraint of the



a—Structure of tough sample consists of light etching outer layer, the ferritic grains of Cr-Fe alloy with some intergranular constituents, and the interfacial zone between the coating and the steel core.

b—Structure of brittle sample consists of the outer layer, the ferritic grains with greater amount of intergranular constituents, and the interfacial layer.

Fig. 2—Micrographs of chromized coatings of brittle and tough samples are shown to illustrate the structural difference between the two types. Samples were etched with Marble's reagent. X1000. Area reduced approximately 55 pct for reproduction.

sample, the embrittled coating is first fractured; then the cracks propagate through the otherwise tough steel core, resulting in complete fracture of the sample.

Compared to the tough coatings, the brittle coatings are thinner, richer in chromium content, and contain greater amount of the light etching constituents. Coating thickness is closely related to the chromium content of the coating and prior carbon content of the steel. The growth of the coating during chromizing is much influenced by the surface reactions and subsequent diffusion rates. Higher carbon content in the steel would result in formation of more carbides and, hence, a more effective diffusion barrier. Retardation of diffusion of the chromium atoms due to the carbide barrier in turn results in a thinner coating of higher chromium concentration.

Heat treatment results show that the higher chromium coatings are readily susceptible to embrittlement by slow cooling through the embrittling range; whereas the lower chromium coatings are little affected, if any, by a similar treatment. This is in line with the observations of other investigators in their study of Cr-Fe alloys.[3] Thus, if diffusion in a nonchromizing atmosphere is resumed, the resulting growth of coating thickness and a lowering of the chromium concentration should reduce the susceptibility to embrittlement. This is well demonstrated by the experimental results.

The outer layer and light etching constituents are believed to be principally a mixture of chromium and iron carbides on the basis of analytical results. These carbides are apparently not associated with the mechanism of embrittlement, as evidenced by

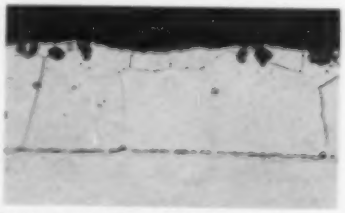


Fig. 3—Micrograph shows the chromized coating of the brittle sample of Fig. 2b after heat treatment at 1150°C for 30 min in hydrogen. Note the absence of the outer layer and the intergranular constituent. The interfacial zone is better resolved and appears to be multilayered. Etched with Marble's reagent. X1000. Area reduced approximately 55 pct for reproduction.

their retention in various parts of the coating after the 700°C treatment and removal of brittleness that followed. According to Fisher, Dulis, and Carroll,[3] the so-called 475°C embrittlement of ferritic Cr-Fe alloys is accompanied by the precipitation of a chromium-rich ferrite phase, probably coherent with the matrix. The presence of this phase may be manifested by a darkening of the matrix under reflection microscope. However, in the present study, such a darkening effect was not noted.

Formation of the outer layer is believed to be associated with both the carbon content of the steel core and the time-temperature relationship of the chromizing process employed. While diffusion is retarded by formation of carbide, the reduction reaction on the surface is not so affected. This situation then leads to accumulation of chromium and carbon on the surface and, hence, the layer of mixed carbides. Since the retarding effect of carbide on diffusion rate is enhanced by lower temperature, it is believed that most of the build-up of this outer layer took place during the early part of slow cooling from the chromizing temperature.

Acknowledgments

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Solubility and Density of Hydrated Aluminas in NaOH Solutions

by Allen S. Russell, Junius D. Edwards, and Cyril S. Taylor

Solubilities and densities are reported for alumina hydrates in NaOH solutions under the conditions of the Bayer process employed to purify alumina for aluminum production. The equilibrium constants for reactions to form AlO_2^- accurately correlate the effects of NaOH concentration on the solubilities; the temperature coefficients of the equilibria lead to heats of solution of 7.34, 4.76, and 5.24 kcal for the α trihydrate, α monohydrate, and β trihydrate, respectively. Moderate calcination does not change the solubility of the α trihydrate but increases that of the α monohydrate. At solution temperatures above 100°C, the trihydrates transform rapidly to α monohydrate. Hydrothermally produced α monohydrate is the stable phase above 20°C.

CORNERSTONE of the aluminum industry, the Bayer process for alumina production is based on the difference in solubility of hydrated alumina in NaOH solutions between 170° and 40°C. Solubilities and densities in this system were determined thirty years ago by M. Tosterud[1] under the direction of J. D. Edwards and C. S. Taylor. The solubility data, which still appear more concordant than those in the literature, are employed to sort out the complex equilibria in this system.

The nomenclature for the alumina hydrates is that presented in *The Aluminum Industry*[2] and more recently summarized in *Alumina Properties*.[3] α trihydrate is the purified phase analogous to gibbsite, α monohydrate is similarly akin to boehmite, and β trihydrate is sometimes known as bayerite.

Literature

Goudriaan[4] reviewed the early literature on alumina in NaOH solutions and presented data on the solubility at 30°C of several forms. The gel precipitated from aluminum sulphate or chloride with ammonia and dried at 130° to 140°C had the highest solubility but was unstable, its solubility being cut to one-tenth on standing in NaOH. The solubility of the gel was decreased one-third on calcination at 300° to 400°C, while that of the gel transformed in NaOH at room temperature to a crystalline trihydrate was unchanged on calcination. The preparation produced by slow hydrolysis of sodium aluminate solution with CO_2 in the air gave solubilities in rough agreement with later values for α trihydrate. The solubility of this form was increased slightly by the calcination.

Fricke and Jucaitis[5] extended the work to 30° and 60°C with α trihydrate. They also gave solubilities for β trihydrate (bayerite b), although the residue belonging to the left branch of the solubility curve corresponded to α trihydrate. The residues at high NaOH concentrations were mono and trisodium aluminates, the latter being $3Na_2O \cdot Al_2O_3 \cdot 6H_2O$.

Solubilities of α trihydrate at 95°C and of α monohydrate at 150° and 200°C come from the work of Magarchak.[6] α trihydrate transformed to α monohydrate above 95°C.

A few measurements on the solubility at 25°C of an alumina trihydrate prepared by slow hydrolysis of $NaAlO_2$ solutions were presented by Sprauer and Pearce.[7] Their analyses showed the monosodium aluminate to have the composition $Na_2O \cdot Al_2O_3 \cdot 2.5H_2O$.

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Bauermeister[8] and Bauermeister and Fulda[9] published solubilities for α trihydrate between 74° and 94°C from parallel experiments in which the alumina concentration was both lower and higher (with seed α trihydrate to induce crystallization) than the solubility limit. Three days were required for adjustment of the solutions to their final states. The loss on ignition of the solid residue at 100°C was that of a trihydrate, but at the higher temperatures it was α monohydrate.

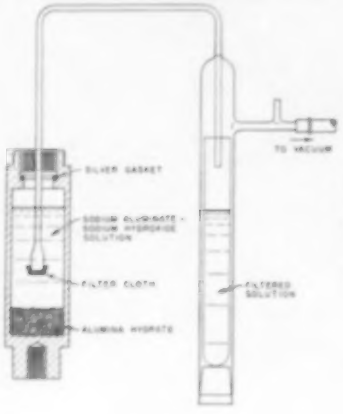

Fig. 1—Diagram shows steel bomb and sampling device used.

Tomonari[10] calculated the equilibrium constant K for the solubility reaction as 0.1 (temperature not stated) for commercial trihydrate in NaOH solution from the relation

$$K = \left(\frac{\text{Concentration of NaAlO}_2}{\text{Concentration of free caustic soda}}\right) \times \left(\frac{\text{Vapor pressure of solution}}{\text{Vapor pressure of water}}\right)^4.$$

Tomonari's presentation prevents exact comparison, but his values agree roughly with those of other workers. Herrmann[11] reported a few solubility de-

Table I. Density, Solubility, and Equilibrium Constant of α Trihydrate in NaOH Solutions

(1) Density, G per Ml	(2) Na_2O, Gpl	(3) Al_2O_3, Gpl	(4) Al_2O_3/ Na_2O	(5) AlO_2^-, Equivalents per L	(6) OH^-, Equivalents per L	(7) H_2O^* Activity	(8) Cols. (5) (7)‡ / (6)
40°C Temperature for 264 Hr†							
1.021	15.5	2.5	0.159	0.048	0.45	0.99	0.104
1.053	38.5	6.0	0.156	0.118	1.12	0.97	0.099
1.096	76.0	14.9	0.200	0.275	1.98	0.92	0.118
1.107	78.0	16.3	0.208	0.318	2.20	0.91	0.120
1.126	92.5	19.7	0.214	0.388	2.60	0.88	0.116
1.163	122	28.4	0.233	0.555	3.38	0.83	0.113
1.198	149	37.9	0.255	0.746	4.06	0.79	0.114
1.292	217	81.2	0.374	1.59	5.41	0.67	0.132
1.371	268	144	0.532	2.79	5.85	0.56	0.148
50°C Temperature for 168 Hr†							
1.021	15.5	3.3	0.218	0.066	0.43	0.99	0.150
1.054	39.0	8.6	0.222	0.161	1.09	0.97	0.138
60°C Temperature for 168 Hr†							
1.020	15.5	4.4	0.281	0.085	0.42	0.99	0.200
1.055	38.5	11.2	0.291	0.211	1.02	0.97	0.194
1.101	70.0	23.6	0.338	0.464	1.79	0.92	0.220
1.113	78.5	27.1	0.346	0.533	1.97	0.91	0.223
1.132	92.0	32.9	0.359	0.648	2.32	0.88	0.216
1.172	120	47.4	0.395	0.921	2.94	0.83	0.219
70°C Temperature for 96 Hr†							
1.022	15.5	5.6	0.360	0.109	0.39	0.99	0.274
1.058	39.0	15.2	0.392	0.291	0.96	0.97	0.292
1.105	70.0	29.5	0.421	0.578	1.68	0.92	0.292
1.117	78.0	33.5	0.432	0.661	1.86	0.91	0.296
1.137	91.5	40.9	0.449	0.806	2.15	0.88	0.291
1.178	120	58.9	0.494	1.16	2.70	0.83	0.301
1.220	149	79.6	0.547	1.58	3.23	0.79	0.304
1.320	205	150	0.727	2.96	3.71	0.69	0.373
1.420	255	233	0.913	4.55	3.68	0.59	0.433
80°C Temperature for 72 Hr†							
1.022	15.5	7.1	0.452	0.138	0.36	0.98	0.373
1.059	38.5	18.9	0.493	0.373	0.97	0.97	0.403
1.110	70.0	37.5	0.536	0.736	1.52	0.92	0.411
1.123	78.0	42.8	0.548	0.836	1.68	0.91	0.415
1.144	92.0	53.2	0.569	1.03	1.94	0.88	0.411
1.186	118	74.0	0.624	1.44	2.35	0.83	0.435
90°C Temperature for 48 Hr†							
1.023	16.0	8.7	0.552	0.173	0.34	0.98	0.494
1.061	38.0	22.2	0.585	0.436	0.79	0.97	0.513
1.114	69.5	44.3	0.632	0.861	1.39	0.92	0.530
1.127	77.5	51.0	0.658	1.00	1.50	0.91	0.547
1.147	90.0	61.0	0.679	1.20	1.71	0.88	0.544
1.192	117	86.0	0.736	1.69	2.09	0.84	0.575
100°C Temperature for 24 Hr†							
1.027	17.0	11.5	0.676	0.226	0.32	0.99	0.69
1.064	38.5	27.1	0.704	0.532	0.71	0.97	0.70
1.181	71.0	53.6	0.755	1.05	1.24	0.92	0.71
110°C Temperature for 5 Hr†							
1.123	60	60	0.87	1.17	1.06	0.92	0.94
1.137	70	69	0.87	1.37	1.20	0.91	0.95
1.158	80	78	0.88	1.54	1.34	0.88	0.99
1.158	80	81	0.90	1.57	1.31	0.88	0.93
1.206	117	111	0.95	2.15	1.54	0.84	0.98
120°C Temperature for 3 Hr†							
1.126	60	63	0.93	1.34	0.95	0.92	1.09
1.150	82	78	0.96	1.54	1.11	0.90	1.12
1.253	139	140	1.08	2.96	1.53	0.80	1.23
1.347	192	233	1.21	4.58	1.64	0.71	1.40
130°C Temperature for 1 Hr†							
1.130	60	69	1.01	1.35	0.85	0.92	1.34
1.143	73	79	1.05	1.55	0.97	0.91	1.47
1.154	81	88	1.08	1.72	0.90	0.90	1.55
1.165	88	98	1.06	1.81	1.03	0.89	1.41
1.1891	100	113	1.13	2.20	1.03	0.87	1.62
1.214	113	127	1.12	2.48	1.16	0.85	1.54
1.256	134	161	1.20	3.16	1.17	0.81	1.78
140°C Temperature for ½ Hr†							
1.131	60	73	1.064	1.42	0.79	0.92	1.5
1.155	81	91	1.124	1.78	0.83	0.90	1.8
1.2181	112	138	1.211	2.66	0.96	0.85	2.0
150°C Temperature for ½ Hr†							
1.131	66	73	1.11	1.44	0.69	0.93	1.8
1.147	75	84	1.12	1.66	0.77	0.91	1.8
1.156	81	94	1.16	1.84	0.77	0.90	2.0
1.167	87	101	1.16	1.98	0.83	0.89	1.9
1.2031	101	126	1.25	2.48	0.78	0.87	2.4
1.222	111	141	1.27	2.77	0.82	0.85	2.5
1.261	139	188	1.30	3.29	0.87	0.82	2.6
160°C Temperature for 1/3 Hr†							
1.135	67	75	1.124	1.47	0.69	0.92	1.8
1.160	89	99	1.241	1.95	0.64	0.90	2.5
170°C Temperature for 1/3 Hr†							
1.134	64	80	1.25	1.57	0.50	0.92	2.7
1.164	89	106	1.32	2.07	0.51	0.90	3.3
1.1811	88	111	1.29	2.18	0.60	0.89	2.9
1.2101	99	124	1.35	2.43	0.77	0.87	2.4
1.222	109	150	1.38	2.95	0.97	0.85	3.8

* Data of Hayward and Perman[14] for NaOH solutions.
† Hours are approximate times for equilibrium.
‡ Density was calculated.
§ Omitted from Fig. 1 for clarity.

terminations in his consideration of the rate of this reaction.

Experimental Methods

An excess of hydrated alumina was added to 50 ml of NaOH solution in a 110 ml steel bomb, which was sealed by a silver gasket, Fig. 1. Four of these bombs were rotated from 5 min to 2 months in an oil bath controlled to 0.2°C. Above 100°C, where the transformation of trihydrate into monohydrate becomes rapid, the change of alumina content of the solution was followed by sampling, Fig. 1, at short intervals of time (for example, every 5 min at 170°C); at lower temperatures, longer intervals were employed. After the mixing, the bombs were rapidly cooled. The cooling had no large effect on the amount of alumina held in solution if the sample was not allowed to stand for any great length of time, as is shown by the following percentages of Al_2O_3 that resulted from various cooling times: 15 min, 3.892 pct; 30 min, 3.904 pct; and 60 min, 3.908 pct.

The weighed 2 to 5 ml filtered sample was diluted to 200 ml, acidified with HCl, heated just to boiling, and dilute NH_4OH added dropwise until methyl red changed to a distinct yellow. The solution was boiled for 2 min, filtered, and the precipitate washed thoroughly with hot 5 pct NH_4Cl. The alumina was next dissolved with HCl, reprecipitated, filtered, and washed as before, dried, and ignited to constant weight.

The NaOH concentration was determined on another sample by titrating the boiling solution with standard HCl until the color of phenolphthalein was discharged. The concentrations are for the final equilibrium solutions and are lower than for the initial solutions because of dilution by water evolved from the hydrated alumina. This titration, of course, gives both the free caustic and that which was combined with the alumina.

The densities were determined for the less viscous solutions by weighing a platinum cylinder of known mass and volume in the liquid sample; for more viscous solutions, a pycnometer was employed. Values originally measured at a known room temperature were converted to 25°C by an equation shown later.

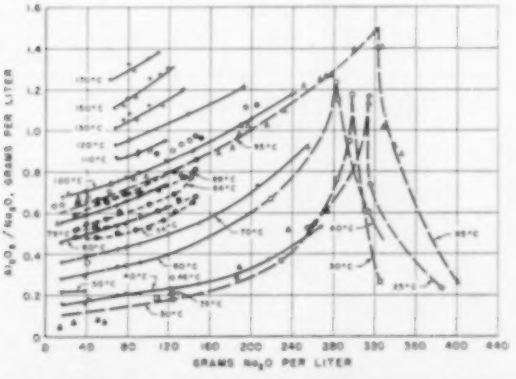

Fig. 2—Solubilities of α trihydrate in NaOH solutions are plotted in comparison with the literature values. Closed triangle represents data of Goudriaan[4] at 30°C; square, Fricke and Jucaitis[5] at 30° and 60°C; open triangle, Magarchak[6] at 95°C; diamond, Spreuer and Pearce[7] at 25°C; closed circle, Bauermeister[8] at 74°, 79°, 84°, 89°, 94°, and 100°C; open circle, Herrmann[11] at 30°, 35°, 40°, 46°, and 100°C; and plus, the authors' work.

Table II. Influence of Variables on Time Required for 85 Pct Transformation of α Trihydrate to α Monohydrate, Judged by Loss on Ignition of Residue

Na_2O Concentration, Gpl	Hr for 85 Pct Conversion	
	150°C	170°C
124	6.0	0.75
83	10.5	3.0

Stick NaOH was purified from alcohol and freed from carbonate by the addition of the requisite amount of $Ba(OH)_2$. The α trihydrate was the regular commercial product washed to free it from excess soda. The α monohydrate was prepared by digesting the trihydrate in 77.5 g Na_2O per l for 5 hr at 170°C. The solid was filtered off, washed with hot water, and dried at 105°C. This gave a uniform product which showed loss on ignition of about 17 pct, compared to the theoretical value of 15.0 pct. To prepare β trihydrate, the α trihydrate was dissolved in hot 10 pct NaOH solution, precipitated by passing CO_2 into the hot solution, filtered, agitated repeatedly in distilled water until the wash water was practically neutral, filtered, and dried in air at 105°C. These procedures are not necessarily the ones which would be preferred today for preparation and analysis of these phases.

Experimental Results

In Table I are shown the densities and maximum amounts of α trihydrate dissolved from 40° to 170°C with solutions containing up to 200 g Na_2O per l. At temperatures below 100°C, the rate of transformation of trihydrate to the less soluble monohydrate is slow, and fairly accurate values for the solubility could be obtained. At higher temperatures, samples were taken at short intervals of time (every 5 min at 170°C) and the maximum values were recorded. It will be evident later that the results above 120°C are probably below the true solubilities. The solubilities expressed in the weight ratio Al_2O_3/Na_2O are plotted in Fig. 2 in comparison with the literature values. Table I also shows that long times are required for equilibrium at the lower temperatures.

The final value for solubility at 100°C and 77.5 g Na_2O per l is identical for 80 to 200 mesh and "through 200 mesh" samples of α trihydrate separated by screening from the commercial hydrate,

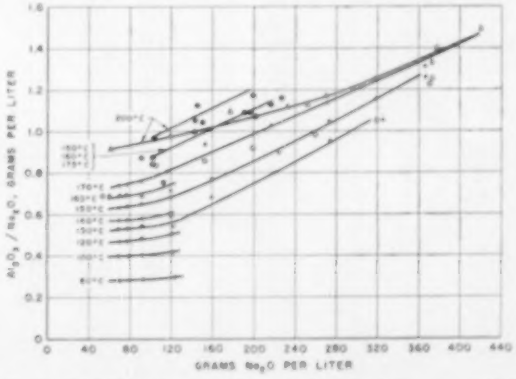

Fig. 3—Solubilities of α monohydrate in NaOH solutions are plotted in comparison with the literature values. Open circle represents the data of Magarchak[6] at 150°C; triangle, Magarchak[6] at 200°C; closed circle, Bauermeister and Fulda,[9] and plus, the authors' work.

although the rate of solution is somewhat faster for the finer sample.

The rate of change of α trihydrate into α monohydrate increases both with temperature and with strength of the NaOH solution. The influence of these variables on the time required for 85 pct transformation in a bomb (judged by loss on ignition of the residue) is given by the experiments summarized in Table II.

The solubility of alumina in Na_2CO_3 solution is very low. Only 0.18 pct Al_2O_3 is dissolved when an excess of α trihydrate is agitated in 100 g Na_2CO_3 per l at a temperature of 90°C for 48 hr. The addition of 40 to 95 g Na_2CO_3 per l at 90°C or of 60 to 93 g Na_2CO_3 per l at 150°C has no measurable effect upon the solubility of α trihydrate.

Table III. Density, Solubility, and Equilibrium Constant of α Monohydrate in NaOH Solutions

(1) Density, G per Ml	(2) Na_2O, Gpl	(3) Al_2O_3, Gpl	(4) Al_2O_3/Na_2O	(5) AlO_2^-, Equivalents per L	(6) OH^-, Equivalents per L	(7) H_2O^* Activity	(8) Calc. (5) (7)‡ (6)
			80°C Temperature for 1008 Hr†				
1.103	70	19.6	0.280	0.385	1.87	0.92	0.189
1.114	80	22.5	0.281	0.441	2.14	0.90	0.186
1.131	93	26.5	0.287	0.520	2.46	0.88	0.186
1.170	124	36.6	0.296	0.718	3.27	0.83	0.182
			100°C Temperature for 360 Hr†				
1.107	70	27.7	0.396	0.543	1.70	0.92	0.294
1.119	79	31.6	0.400	0.620	1.93	0.90	0.289
1.138	92	37.1	0.403	0.728	2.24	0.88	0.286
1.176	122	51.5	0.422	1.01	2.93	0.83	0.286
			120°C Temperature for 120 Hr†				
1.105	70	32.6	0.465	0.640	1.62	0.92	0.363
1.118	78	36.8	0.472	0.722	1.79	0.90	0.363
1.136	92	44.6	0.487	0.875	2.08	0.88	0.370
1.176	121	61.6	0.505	1.21	2.70	0.83	0.372
			130°C Temperature for 70 Hr†				
1.1091	69	36.9	0.534	0.724	1.51	0.92	0.44
1.1221	78	41.3	0.530	0.810	1.71	0.90	0.43
1.143	92	49.7	0.543	0.975	1.98	0.89	0.44
1.1441	92	50.4	0.548	0.988	1.98	0.88	0.44
1.1911	121	65.6	0.542	1.29	2.61	0.83	0.41
1.255	160	108.6	0.680	2.13	3.01	0.78	0.56
1.355	221	176.6	0.796	3.46	3.66	0.66	0.62
1.448	274	260.1	0.954	5.10	3.73	0.56	0.77
1.538	325	342.2	1.048	6.71	3.77	0.46	0.82
			140°C Temperature for 48 Hr†				
1.1131	70	40.2	0.574	0.788	1.49	0.92	0.49
1.1241	77	44.1	0.572	0.866	1.62	0.90	0.48
1.1471	92	53.4	0.580	1.05	1.92	0.88	0.49
1.1921	120	71.4	0.595	1.40	2.47	0.83	0.47
			150°C Temperature for 24 Hr†				
1.114	70	44.2	0.631	0.867	1.39	0.92	0.57
1.126	78	49.5	0.630	0.971	1.53	0.90	0.57
1.147	92	59.5	0.650	1.17	1.79	0.88	0.57
1.148	91	59.6	0.653	1.17	1.77	0.89	0.58
1.180	120	81.8	0.682	1.60	2.27	0.83	0.58
1.281	159	123.6	0.773	2.43	2.69	0.78	0.71
1.370	224	200.4	0.894	3.93	3.31	0.65	0.77
1.464	274	286.7	1.047	5.63	3.20	0.56	0.99
1.552	319	369.2	1.158	7.24	3.05	0.47	1.12
1.631	366	460.3	1.258	9.03	2.78	0.38	1.24
			160°C Temperature for 24 Hr†				
1.117‡	70	48.5	0.692	0.951	1.31	0.92	0.67
1.129‡	77	53.5	0.695	1.05	1.44	0.90	0.66
1.152‡	92	63.0	0.685	1.24	1.73	0.88	0.63
1.1951	120	86.2	0.718	1.69	2.18	0.83	0.64
			170°C Temperature for 24 Hr†				
1.117	70	51.9	0.741	1.02	1.34	0.92	0.76
1.133	78	58.2	0.746	1.14	1.38	0.90	0.74
1.153	91	68.8	0.756	1.35	1.59	0.88	0.75
1.154	91	70.2	0.756	1.38	1.56	0.89	0.79
1.189	116	93.7	0.808	1.84	1.90	0.83	0.80
1.262	153	143.0	0.835	2.81	2.13	0.78	1.03
1.374	219	224.3	1.026	4.40	2.65	0.66	1.10
1.470	271	308.3	1.130	6.08	2.70	0.56	1.26
1.560	320	394.3	1.240	7.74	2.57	0.46	1.38
1.641	365	477.7	1.308	9.37	2.40	0.38	1.48

* Data of Hayward and Perman[14] for NaOH solutions.
† Hours are approximate times for equilibrium.
‡ Density was calculated.

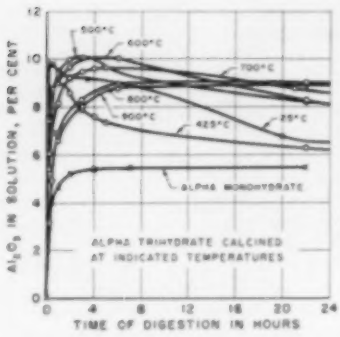

Fig. 4—Effect of time is shown on the rate of solution in NaOH of α trihydrate heated to various temperatures. Pseudo-equilibrium solubility in 90 g Na_2O per l at 150°C.

The solubility of the α monohydrate in solutions ranging up to 365 g Na_2O per l from 80° to 170°C is much lower than that of the α trihydrate under the same conditions. Table III gives the densities and solubilities. Fig. 3 shows the solubility expressed in the weight ratio Al_2O_3/Na_2O as a function of NaOH concentration for various temperatures in comparison with literature values. The time for establishment of equilibrium is much greater at a given temperature for the α monohydrate than for the α trihydrate and only falls to 15 to 30 min at 200°C at 100 g Na_2O per l.

The solubility of the β trihydrate is substantially greater than that of the α trihydrate at 40°C but is nearly the same at 100°C. See Table IV for the limited solubility and density data for this phase.

Moderate calcination of α trihydrate in air, whereby the X-ray indicates a variety of structures,[12] does not produce a major change in the pseudo-equilibrium solubility in 93 g Na_2O per l at 150°C. This calcination, however, causes a decrease in the rates both of solution and of transformation to the stable α monohydrate phase. The effect of

Table IV. Density, Solubility and Equilibrium Constant of β Trihydrate in NaOH Solutions

(1) Density, G per Ml	(2) Na_2O, Gpl	(3) Al_2O_3, Gpl	(4) Al_2O_3/Na_2O	(5) AlO_2^-, Equivalents per L	(6) OH^-, Equivalents per L	(7) H_2O^* Activity	(8) Calc. (5) (7)‡ (6)
			40°C Temperature for 168 Hr†				
1.021	15.1	4.8	0.32	0.095	0.39	0.99	0.24
1.056	35.9	10.1	0.28	0.198	0.96	0.97	0.19
			50°C Temperature for 120 Hr†				
1.021	14.8	6.1	0.41	0.119	0.36	0.99	0.33
1.056	36.0	13.0	0.36	0.255	0.91	0.97	0.26
			60°C Temperature for 96 Hr†				
1.021	14.7	6.8	0.46	0.133	0.34	0.99	0.38
1.057	35.8	15.0	0.42	0.294	0.85	0.97	0.32
			70°C Temperature for 48 Hr†				
1.022	15.0	8.0	0.53	0.156	0.33	0.99	0.47
1.060	35.2	18.3	0.52	0.359	0.78	0.97	0.44
			80°C Temperature for 16 Hr†				
1.021	15.1	9.2	0.61	0.181	0.31	0.99	0.58
1.060	35.2	21.8	0.62	0.428	0.71	0.97	0.57
			90°C Temperature for 3½ Hr†				
1.020	15.0	10.5	0.70	0.206	0.28	0.99	0.73
1.060	35.1	25.3	0.72	0.496	0.64	0.97	0.73
			100°C Temperature for 5½ Hr†				
1.020	16.0	12.8	0.80	0.251	0.27	0.99	0.88
1.063	35.5	29.1	0.82	0.571	0.57	0.97	0.94

* Data of Hayward and Perman[14] for NaOH solutions.
† Hours are approximate times for equilibrium.

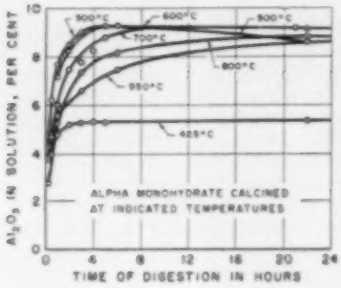

Fig. 5—Effect of time is shown on the rate of solution in NaOH of α monohydrate heated to various temperatures. Pseudo-equilibrium solubility in 90 g Na_2O per l at 150°C.

time on the solubility of these calcined products is shown in Fig. 4. Additional determinations of solubilities, at the times shown to produce maximum values, are listed in Table V. The hydration of the residues toward monohydrate is evident from the loss on ignition values.

Calcination of the α monohydrate at 500° to 600°C, in contrast to the α trihydrate, increases both the amount and rate of the solubility. Calcination temperatures below 425°C are without effect and higher temperatures decrease the rate of solution. The solubility of the calcined monohydrate decreases upon long digestion as the monohydrate is formed again. These effects are shown in Fig. 5 and additional data are given in Table VI.

Table V. Solubility of NaOH Solutions at 150°C of Partially Calcined α Trihydrate

Calcination, 1 Hr at °C	Ignition Loss, Pct	Time of Solution, Hr	Density, G per Ml	Na_2O, Gpl	Al_2O_3 Pct	Al_2O_3 Gpl	Al_2O_3/Na_2O	Ignition Loss of Residue, Pct
None	34.6	½	1.167	87	8.7	101	1.16	—
300	10.0	¼	1.178	86.5	9.1	107	1.21	15.4
500	2.8	2½	1.181	88.2	9.6	114	1.29	6.1
500	2.8	3	1.184	91.0	9.9	117	1.29	7.3
600	1.6	4	1.178	89.3	9.4	110	1.23	5.8
700	1.6	8	1.178	89.7	9.7	114	1.27	3.2

α trihydrate heated at 1140°C, under conditions such that it undergoes no exothermal change (a γ-type alumina), dissolves at 150°C in 93 g Na_2O per l to the same extent in 65 hr as the uncalcined hydrate in 15 min; thereafter its solubility decreases slowly while it rehydrates to 8.17 pct loss on ignition after 138 hr. The same material, heated in the presence of a small amount of a fluoride to an exothermal transformation at 1092°C (α alumina), has only one-quarter as much solubility in 164 hr and rehydrates in this time only to 0.28 pct. The hydrothermally formed α monohydrate gives similar results under corresponding calcination.

Density of $NaAlO_2$ Solutions: The densities d in grams per milliliter for solutions of NaOH saturated at T °C with α trihydrate (at least for concentrations up to 200 g Na_2O per l) can be expressed by the formula

$$d = 1.002 + (0.00109 + 5.9\times10^{-6}T) \times (\text{g } Na_2O \text{ per l})$$

or by its alternate form

$$d = 1.002/[1 - (0.0109 + 5.9\times10^{-4}T) \times (\text{pct } Na_2O)].$$

The equations for solutions saturated with hydrothermally produced α monohydrate are

$$d = 1.002 + (0.00114 + 3.1\times10^{-6}T) \times (\text{g } Na_2O \text{ per l})$$

$$d = 1.002/[1 - (0.0114 + 3.1\times10^{-4}T) \times (\text{pct } Na_2O)].$$

Other formulas which are useful for estimation of densities are

$$d = 1.002 + 0.00125 \times (\text{g } Na_2O \text{ per l}) + 0.0005 \times (\text{g } Al_2O_3 \text{ per l})$$

$$d = 1.002/(1 - 0.0125 \times (\text{pct } Na_2O) - 0.0005 \times (\text{pct } Al_2O_3).$$

The densities of a number of representative solutions, beside being determined at a definite room temperature, were also determined at a measured elevated temperature in the vicinity of 80°C. The equations representing the change in density with change in temperature of measurement are as follows

$$d_t = d_{80} - (T\text{-}25) \times \alpha$$

where α is 0.00075 for 75 to 100 g Na_2O per l and 0.00077 for 100 to 125 g Na_2O per l, both solutions being saturated with alumina.

Hydrolysis Equilibria

For the reactions

$$Al(OH)_3 + OH^- \rightarrow AlO_2^- + 2H_2O$$

$$AlOOH + OH^- \rightarrow AlO_2^- + H_2O$$

the equilibrium constants K for the case that solid alumina phases are present and the activity coefficients of the OH^- ions and AlO_2^- ions are equal, are

$$K_3 = \frac{C_{AlO_2^-} \times A_{H_2O}^2}{C_{OH^-}}$$

$$K_1 = \frac{C_{AlO_2^-} \times A_{H_2O}}{C_{OH^-}}$$

where $C_{AlO_2^-}$ and C_{OH^-} are the equivalents per liter of aluminate and hydroxyl ions and A_{H_2O} is the activity of the water.

Hayward and Perman[14] measured the vapor pressure of water over NaOH solutions from 30° to 80°C and at concentrations up to 450 g Na_2O per l, and Robinson and Stokes[15] reported values at 25°C over a range of concentration. The use of these values for the activity of water in Tables I, III, and IV assumes that they are independent of temperature and of the amount of alumina dissolved in the solutions. The last columns of Tables I, III, and IV show

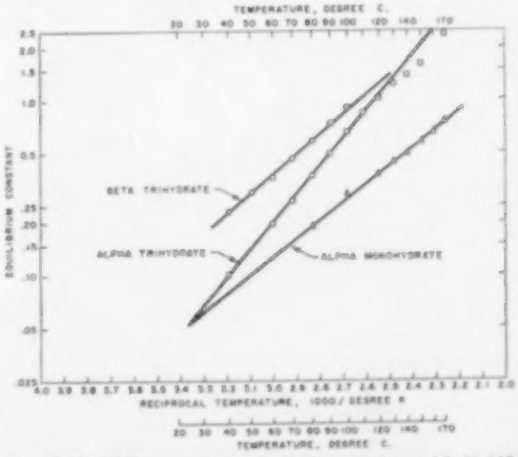

Fig. 6—Equilibrium constants of alumina reactions with $NaAlO_2$ solutions are plotted vs temperature.

Table VI. Solubility in NaOH Solutions at 150°C of Partially Calcined α Monohydrate

Calcination, 1 Hr at °C	Ignition Loss, Pct	Time of Solution, Hr	Density, G per Ml	Na_2O, Gpl	Al_2O_3, Pct	Al_2O_3, Gpl	Al_2O_3/Na_2O	Ignition Loss of Residue, Pct
None	17	24	1.148	92	5.18	59.5	0.65	—
500	3.1	4½	1.177	91	8.79	103	1.14	6.3
500	3.1	5	1.181	92	9.25	109	1.19	6.4
600	1.7	6	1.180	89	9.35	110	1.24	6.2
700	0.8	6	1.177	91	8.68	102	1.12	4.0
700	0.6	7	1.178	91	9.09	105	1.18	—

the equilibrium constants which are calculated from the authors' solubility data and this water activity. In calculating the concentration of OH^-, the measured hydroxyl concentration is corrected for the formation of aluminate ion. Inasmuch as the resulting values increase with NaOH concentration (presumably because of the failure of the foregoing assumptions at higher concentrations), values of the equilibrium constant at the various temperatures are derived by extrapolation to 0 NaOH concentration. Plots of the equilibrium constants as a function of temperature are shown in Fig. 6 for the three phases.

Essentially the same values for the equilibrium constant are derived from the assumption that the activity of the water is the square of its mol fraction. In deriving the water concentration, the weight of NaOH and of $NaAlO_2$ is subtracted from the total weight of a given volume of solution, and the result is divided by the molecular weight of water. The mol fraction of the water is then the mols of water divided by the sum of the mols of water plus the total number of equivalents of sodium, aluminate, and hydroxyl ions in solution.

The equations representing the solubility of these phases are then (T in °K) as follows: for α trihydrate

$$\log K_a = \frac{-7340}{4.574T} + 4.11$$

for α monohydrate

$$\log K_\alpha = \frac{-4760}{4.574T} + 2.22$$

for β trihydrate

$$\log K_a = \frac{-5240}{4.574T} + 3.01.$$

Heats of hydrolysis from these data are 7.34 kcal for α trihydrate, 4.76 kcal for α monohydrate, and 5.24 kcal for β trihydrate. *Selected Values*[14] lead to 6.57 and 3.54 kcal for the α trihydrate and α monohydrate, respectively.

For either the α trihydrate or hydrothermal α monohydrate, the equilibrium constant at room temperature is about 0.05. From the relation between this constant and the ion product of water, 10^{-14}, the solubility product of the monobasic aluminum acid, $HAlO_2$, is $(AlO_2^-) \times (H^+) = 2 \times 10^{-13}$, a value in agreement with that of Lacroix.[15]

Transition Temperatures

The expression for the transition temperature from α trihydrate to α monohydrate is

$$\frac{7340 - 4760}{4.574(4.11 - 2.22) - \log A_{H_2O}}.$$

The calculated transition temperatures are 27°C for 0 NaOH concentration, 25°C for 80 g Na_2O per l (0.9 activity H_2O), 23°C for 140 g Na_2O per l (0.8 activity H_2O), etc. The β trihydrate is unstable with respect to α trihydrate at temperatures below 145°C.

The stabilities shown by this work for the hydrated alumina phases are difficult to reconcile with other experience. α trihydrate is produced in the Bayer process, although the hydrothermal-type α monohydrate is the stable phase. Even more confusing is the fact that freshly precipitated α monohydrate left in alkaline solution at room temperature may gradually transform to β trihydrate and then finally to α trihydrate. However, the coarse α monohydrate produced hydrothermally is not converted to α trihydrate on standing in dilute alkali at room temperature. Two factors may provide the explanation: 1—the transformation of α trihydrate to hydrothermal-type α monohydrate is extremely sluggish at room temperature and 2—the α monohydrate form usually produced by precipitation is much more soluble than that produced hydrothermally. Evidence on this latter point is in the work of Goudriaan quoted earlier. It should also be remembered that α monohydrate is produced by moderate heating of α trihydrate and that partially calcined α trihydrate is as soluble as the uncalcined trihydrate. As a matter of fact, the curve for α trihydrate calcined to 425°C, Fig. 4, which shows a decreasing solubility with increasing time of digestion might well result from a change from α monohydrate (from calcination) to α monohydrate (hydrothermal). Other evidence that all the phases which show α monohydrate to the X-ray are not equivalent has been shown by the compilation of Russell.[4]

Discussion of this paper, if any, will appear in JOURNAL OF METALS, November 1956, and in AIME Metals Branch *Transactions*, Vol. **206**, 1956.

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Oxidation of Phosphorus and Manganese During and After Flushing in the Basic Open Hearth

by John F. Elliott and Frank W. Luerssen

FLUSHING the early slag from a stationary open hearth having a high percentage of hot metal in its charge is necessary in order to remove silica from the system. The flush slag is strongly oxidizing and is somewhat acidic. It has, however, considerable capacity to extract phosphorus from the bath and it also removes considerable manganese. It seems probable that factors which control the distribution of phosphorus and manganese between slag and metal in the refining period also should be dominant in the flush and postflush periods. Several studies, as summarized elsewhere,[1, 2] support the viewpoint that conditions closely approaching equilibrium for these elements are rather readily established during the refining period. Over the years these studies have repeatedly demonstrated that 1—high slag volume, 2—low bath and slag temperature, 3—basic slag, and 4—strongly oxidizing slag favor rapid elimination of phosphorus from the bath to the slag. They also show that the following conditions favor retention of manganese in the bath: 1—low slag volume, 2—high bath and slag temperature, 3—basic slag, and 4—minimum oxidizing power of slag. When it is considered that the flush slag often carries as high as 75 pct of the manganese charged and only 25 to 60 pct of the phosphorus charged, it is evident that in removing silica much manganese is sacrificed but phosphorus removal is far from complete. Because of overriding circumstances, this is accepted in most operations and actually it is considered to be inevitable. This may account for the fact that little attention has been paid to conditions affecting the elimination of phosphorus and manganese in the flush slag. A recent study of the behavior of various charge oxides has developed considerable information on the flush and postflush periods. Because the data are felt to be of general interest, they have been brought together and presented in this paper. The object is to show the various factors in the flush and postflush periods which influence elimination of phosphorus and manganese.

Physical Conditions During and After Flushing

Physical conditions existing during the flush vary from plant to plant, from shop to shop, from furnace to furnace, and even from heat to heat. They are strongly influenced by the physical and chemical character of the charge oxide which is ordinarily necessary to provide sufficient oxidizing power early in the heat. Invariably the period is characterized by a vigorous reaction between the principal reactants: the hot metal being added and the charge oxide. During the flush, it is probable that the slag acts to some extent as an oxidizer; but, because of the critical influence of the behavior of the charge oxide on flushing action, it seems apparent that the oxide itself is the dominant oxidizer. Fig. 1 shows the course of two heats which were selected as being typical of the group studied. Heat *A* was charged with 55 pct hot metal, based on the total metallics charged, and heat *B* had 57 pct hot metal. As indicated in Table I and Fig. 1, the melt-down slag, which is not usually voluminous and which is principally FeO, expands greatly in volume and will show rather high levels of SiO_2, MnO, and P_2O_5 very soon after the beginning of the hot metal addition. Simultaneously, large volumes of CO are liberated which cause violent mixing of slag and metal. It is of interest to note that the time required to bring carbon down to a low level is very much longer than that required for the removal of silicon, manganese, or phosphorus. At the end of flush, carbon in the bath is still approximately 2 pct.

When strongly reducing hot metal is brought into contact with strongly oxidizing conditions within the furnace, it is probable that the rate of mass transfer to the slag (and atmosphere) of silicon, manganese, phosphorus, and carbon initially depends principally on the rates at which the two participating phases are brought into contact That is, it depends on the nature of the various reactions. Later in the flush period, when the scrap is virtually all dissolved and the action of the bath has settled down to a steady and somewhat gentle boil, it is likely that other factors, such as the transfer of oxygen across the slag-metal interface, become dominant. The temperature of the slag-metal system is far from uniform. Heat is being driven by the flame down through the slag. Bubbling and surging of the metal also frequently brings portions of the bath in contact with the flame. At areas of contact between the ore and liquid metal, or slag and liquid metal, the oxidizing reactions generate much heat. On the other hand, scrap is being melted which tends to absorb large quantities of heat. Because the liquid bath is high in carbon, the steel scrap is brought into solution rapidly. This can proceed at a rather low temperature; and until much of the scrap has been taken into solution, the bath temperature would not be expected to increase appreciably. Consideration of these factors leads to the conclusion that during the flush period the slag should be rather hot and the bath relatively cold. Both observation and temperature measurements bear this out.

Experimental Data

The extended program of charge oxide evaluation permitted study of the widely varying conditions existing during the flushing period. Slag and metal analyses and bath temperatures reported herein (Tables I and II) were obtained toward the latter portion of the work. Four different types of charge oxide, sinter, two types of hydraulic cement-bonded soft ores, and a pyrobonded agglomerate were used in the study. Although the heats reported were from only one 205 ton furnace, they show variations in flush slag analyses all the way from 25 pct FeO, which is typical with the use of a hard natural charge ore, to 45 pct FeO which resulted when a very poorly agglomerated fine ore was used. The physical behavior of the flushes showed a correspondingly wide variation from well controlled reactions to violent surges following periods of inac-

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Table I. Flush Slag Composition

Heat	Slag	Time Test Taken, Min from Closing Flush Hole	Slag Analysis, Wt Pct							
			P_2O_5	MnO	FeO	Fe_2O_3	CaO	SiO_2	MgO	Al_2O_3
C	Early flush	—	2.54	14.9	34.7	2.00	12.1	26.5	5.1	3.9
	Composite flush	—	3.25	14.0	33.8	3.29	13.9	24.4	5.1	3.9
	Late flush	0	6.20	9.61	31.8	4.43	18.7	19.5	5.1	3.5
	Postflush	5	7.68	9.60	26.8	1.72	21.8	21.0	7.7	4.1
	Postflush	24	5.04	6.78	20.3	2.14	24.6	22.0	7.7	4.1
D	Early flush	—	1.64	13.6	31.5	4.0	14.6	25.3	3.8	3.8
	Composite flush	—	2.02	14.8	28.5	2.4	14.6	28.7	4.0	3.8
	Late flush	0	3.47	14.5	26.8	1.6	18.3	28.1	5.2	4.0
	Postflush	30	2.66	13.0	9.3	1.1	27.7	29.9	10.5	5.5
E	Early flush	—	2.89	10.1	39.6	2.43	12.0	37.0	4.9	4.2
	Composite flush	—	2.60	9.68	43.6	2.30	10.5	33.18	5.0	4.3
	Late flush	−15	4.30	8.13	44.6	3.86	10.9	19.20	3.7	3.8
	Late flush	0	1.86	7.10	49.1	5.40	11.6	25.28	3.7	4.5
	Postflush	10	4.60	6.26	36.9	2.00	14.3	21.78	5.4	4.6
F	Early flush	—	1.74	7.35	49.3	0.87	8.1	16.24	4.9	4.2
	Composite flush	—	1.89	6.97	46.8	15.0	8.3	17.04	5.0	4.3
	Late flush	0	2.22	7.38	50.8	7.29	7.6	18.38	3.7	4.5
	Postflush	5	3.45	8.20	48.1	3.43	9.5	18.80	3.4	3.9
	Postflush	20	3.83	7.87	44.8	4.29	8.9	19.56	4.4	4.0
G	Early flush	—	2.74	12.8	33.3	3.3	12.5	25.3	4.9	4.2
	Composite flush	—	3.50	13.4	33.3	2.87	13.5	25.0	5.0	4.3
	Late flush	0	4.33	13.6	31.3	3.15	16.4	20.3	5.7	4.4
	Postflush	27	3.98	10.9	17.5	1.14	34.5	23.1	7.8	6.3

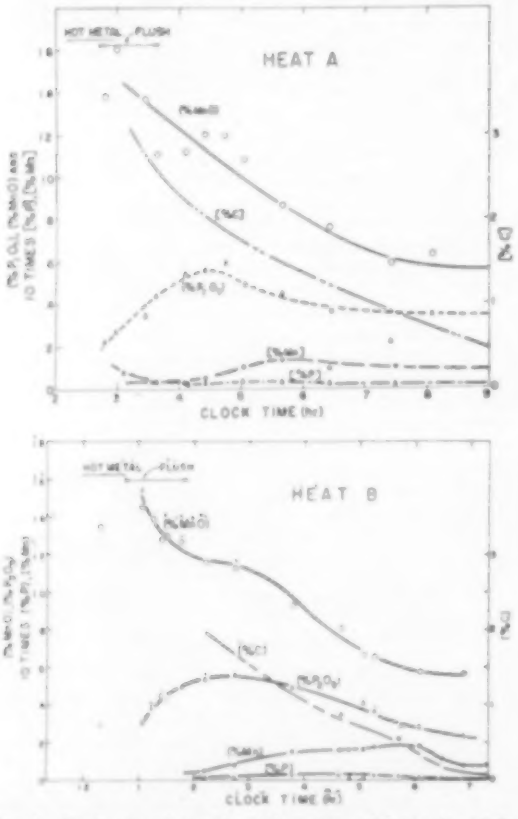

Fig. 1—Graphs give slag and metal analyses of heats A and B during and after flush periods. ( ) represents slag components and [ ] metal components.

tivity. The unusually high levels of MgO in some of the postflush slags probably resulted from bottom erosion. This condition was typical of a heat which had a highly erratic flushing action and the behavior was particularly characteristic of heats using the pyrobonded agglomerate.

Flush slag samples were dipped from the runner and each flush slag analysis reported in Table I was obtained from a composite of five to seven tests taken during the flush. Postflush slag samples were dipped with a conventional spoon from the furnace, usually at the door through which the hot metal was poured. Bath tests were taken almost simultaneously with the slag samples. Each spoon test was killed with high purity aluminum wire after the slag had been pushed away from the center of the spoon. The test was poured in a steel box. Tests showing slag or more than 0.002 pct Si (nil) were discarded. No reliable bath analyses were obtained until very late in the flush because of the lack of complete mixing. Careful analytical work ensured an accuracy of two significant figures in the manganese determinations.

Temperatures: Temperatures of the bath for both postflush and tap periods were carefully obtained with a conventional immersion Pt—Pt-10 pct Rh thermocouple. The potential of the thermocouple was measured with a small portable potentiometer. The temperature read is estimated to have been within ±20°F of the temperature of the liquid in the vicinity of the thermocouple tip. Temperatures were obtained during and after flush and just before the lime boil began. This was done as safety permitted, which usually was 5 to 15 min after closing the flush hole. The flush hole was closed as soon as the flow of slag was reduced to a dribble. On several heats, some slag was flushed as a result of subsequent reactions. As shown in Table II, another set of postflush temperatures and slag-bath samples were obtained on several heats between approximately 15 and 45 min after the end of flush.

After the test program had been completed, review of the data indicated that the bath temperatures for the series did not permit satisfactory interpretation of the data. As a result, slag and bath temperatures were obtained on several additional heats on the same furnace and, as a matter of interest, on a 250 ton unit; see Table III. The temperature distribution in the slag and metal layers in the postflush period when the bath action had settled down to a gentle boil is approximately as shown in Fig. 2.

At this point, it may be of interest to note a few of the observations made during the temperature

surveys. At times when it was possible to get a thermocouple into the bath while the system was in very violent agitation, it was noted that there appeared to be no sharp change in temperature in the vicinity of the slag-metal interface and actually there was a 100° to 450°F difference between the slag and metal. Measurements of slag temperatures were most difficult immediately after hot metal because the wild action in the bath made it impossible to hold the thermocouple at a constant level in the slag as the temperature was taken. Bath temperatures were relatively simple to measure in areas where there were no visible scrap boils. On the other hand, it was obvious at times that the mixing was so violent that there was no regular and clear separation of the liquids. By slowly letting the thermocouple tip sink gradually through the slag and metal layers, it was possible to get an indication of the temperature gradients between the two phases and within each phase. Fig. 3 shows one trace. Table III reports several measurements which appeared typical.

In general, even with a skull on the bottom, the thermal gradient in the bath was 10°F or less. The gradient in the slag layer to within 1 or 2 in. of the top surface was approximately 35° to 70°F. Where larger gradients are shown in the table, it appears that violent mixing of the slag and metal prevented a clear interpretation of the location of the interface. It seems from these data that the large temperature difference observed between the slag and metal probably was confined to the interface between the two phases.

Even toward the end of flush, the metal bath was about the same temperature as that of the hot metal added. The data collected were not sufficiently complete to show whether or not the bath temperatures were influenced by the temperature of the hot metal. A correlation is to be expected. However, it seems certain that other factors such as the degree of meltdown before hot metal is added will be of considerable importance also.

The slag gains temperature much more rapidly than does the metal because: 1—the liquid products from the strongly exothermic reactions go quickly into the slag, 2—the slag is directly exposed to the flame, 3—the slag has a much lower total heat capacity than the bath, and 4—heat is being absorbed from the bath while scrap is being brought into solution at a relatively low temperature by the high carbon iron. Later, the lime boil will tend to keep down the temperature in both the bath and the slag.

Table II. Slag and Metals Analyses for Experimental Charge Oxide

Slag Designation	Bath Temperature, °F	Analysis, Wt Pct												
		C	Mn	P	S	Si	FeO	Fe_2O_3	MnO	P_2O_5	CaO	SiO_2	Al_2O_3*	MgO*
Flush†														
1F‡	2510	1.78	0.012	0.093	0.037	0.002	28.1	2.43	11.7	4.18	16.7	23.30	4.1	7.7
6F	2400	2.62	0.018	0.046	0.040	0.002	27.4	2.29	10.5	6.71	20.3	21.12	4.1	7.7
7F	2455	2.59	0.023	0.062	0.035	0.002	23.5	2.4	12.1	5.65	21.8	25.38	4.1	7.7
8F	2430	2.32	0.020	0.050	0.040	0.002	28.9	2.86	11.1	5.29	16.8	20.98	4.1	7.7
9F	2405	2.64	0.017	0.044	0.038	0.002	26.8	1.72	9.68	7.68	21.8	21.02	4.1	7.7
16F	2410	2.66	0.025	0.081	0.036	0.002	48.1	3.43	8.20	3.45	9.5	18.80	3.0	5.9
17F	2405	2.79	0.014	0.055	0.044	0.002	44.6	3.86	8.13	4.30	10.9	19.20	3.69	3.79
21F	2560	1.71	0.097	0.109	0.033	0.002	13.4	1.3	21.4	3.44	21.7	28.87	3.17	6.26
Postflush†														
2P‡	2430	2.46	0.032	0.047	0.047	0.002	28.9	2.14	14.2	2.69	14.5	26.4	4.1	7.7
3P	2460	2.18	0.011	0.078	0.035	0.002	37.9	1.43	12.5	4.14	10.2	22.68	4.1	7.7
4P	2500	1.85	0.016	0.035	0.051	0.002	30.3	2.72	8.9	4.03	17.6	22.48	4.1	7.7
5P	2505	2.09	0.023	0.028	0.041	0.002	28.5	3.58	9.80	3.96	19.5	21.02	4.1	7.7
9P	2465	2.31	0.019	0.034	0.039	0.002	20.2	2.14	6.78	5.04	24.6	22.02	4.1	7.7
11P	2555	—	0.076	0.018	—	0.002	17.5	1.14	10.9	3.96	24.5	23.12	2.85	15.08
12P	2510	—	0.032	0.031	—	0.002	12.8	0.86	11.2	3.87	26.4	25.38	2.85	15.08
13P	2530	1.63	0.056	0.084	0.037	0.002	9.2	1.1	13.00	2.66	27.7	29.86	5.46	10.50
14P	2580	2.20	0.021	0.041	0.046	0.002	24.9	3.01	10.38	4.56	20.9	22.22	2.85	11.18
15P	2615	1.30	0.031	0.110	0.049	0.002	11.48	1.00	9.47	2.78	25.6	29.21	2.85	16.08
16P	2430	2.50	0.015	0.066	0.040	0.002	44.8	4.29	7.87	3.83	8.9	19.58	3.8	6.9
17P	2470	2.44	0.014	0.045	0.044	0.002	36.9	2.00	8.26	4.65	14.3	21.78	5.38	4.64
19P	2490	—	0.016	0.040	—	0.002	31.0	4.43	9.80	5.40	16.9	19.66	4.50	8.50
20P	2400	2.24	0.019	0.044	0.055	0.002	30.6	2.72	10.1	5.34	17.6	19.97	4.80	6.87
22P	2480	—	0.016	0.048	—	0.002	19.9	1.43	10.5	3.01	23.8	25.83	2.85	15.08
23P	2595	—	0.033	0.090	—	0.002	12.0	1.30	11.0	3.08	27.0	27.52	9.0	9.44
24P	2510	—	0.024	0.042	—	0.002	16.4	1.3	11.1	4.51	27.6	25.16	4.64	9.28
25P	2490	—	0.033	0.037	—	0.002	19.0	1.72	11.91	4.67	23.6	24.70	4.80	9.60
26P	2505	2.06	0.021	0.037	0.037	0.002	25.93	3.58	9.29	5.59	21.9	20.04	5.46	10.50
Refining														
$1R_1$	2740	0.28	0.052	0.009	0.033	0.002	20.1	7.73	2.9	3.38	43.0	11.34	2.5	10.3
$1R_2$	2890	0.05	0.030	0.006	0.026	0.002	26.83	8.01	2.39	2.57	36.7	8.98	2.5	10.3
2R	2890	0.18	0.065	0.008	0.040	0.002	20.50	7.15	4.58	2.82	38.9	9.78	2.5	10.3
$3R_1$	2765	0.15	0.092	0.009	0.033	0.002	12.6	5.0	4.13	4.16	46.8	12.62	2.5	10.3
$3R_2$	2900	0.08	0.070	0.009	0.030	0.002	25.8	7.44	4.32	2.58	36.5	8.90	2.5	10.3
4R	2735	0.22	0.042	0.006	0.039	0.002	21.00	9.45	3.22	2.36	40.4	12.06	2.5	10.3
5R	2825	0.16	0.075	0.009	0.040	0.002	16.6	4.72	3.80	3.58	44.3	15.26	2.5	10.3
6R	2755	0.40	0.068	0.012	0.035	0.001	13.6	5.43	3.16	3.26	45.5	13.18	2.5	10.3
$6R_2$	2920	0.08	0.039	0.010	0.029	0.002	27.48	9.8	2.26	1.67	38.6	7.92	2.5	10.3
8R	2810	—	0.081	0.019	—	0.002	14.3	5.15	4.20	2.85	38.6	16.96	2.5	10.3
$8R_2$	2947	0.05	0.042	0.009	0.040	0.002	22.32	7.58	3.42	2.15	35.9	12.73	2.5	10.3
9R	2860	0.08	0.046	0.007	0.034	0.002	18.10	5.72	3.30	2.75	41.6	14.65	2.5	10.3
$9R_2$	2930	0.07	0.040	0.008	0.033	0.002	22.58	8.46	3.16	2.08	37.7	12.45	2.5	10.3
10R	2825	—	0.033	0.007	—	0.002	25.20	8.20	3.10	1.88	40.3	11.36	3.00	12.90
11R	2935	—	0.050	0.007	—	0.002	24.8	8.9	3.87	1.74	35.7	12.72	5.43	10.00
12R	2840	—	0.06	—	—	0.002	24.6	7.15	3.16	1.86	39.8	12.82	3.00	17.16
$12R_2$	2870	—	0.058	0.007	—	0.002	24.9	7.9	3.48	1.75	37.7	12.82	5.43	10.00
13R	2830	0.06	0.038	0.008	0.032	0.002	23.1	6.9	3.9	2.90	37.9	13.38	3.44	9.01
14R	2925	0.08	0.03	0.006	0.034	0.002	25.48	8.94	2.71	1.72	36.4	11.24	1.78	17.10
15R	2910	0.06	0.025	0.008	0.033	0.002	31.8	12.4	2.7	2.08	32.7	7.60	1.78	6.02
18R	2870	—	0.045	0.009	—	0.002	24.8	9.9	4.1	2.92	38.9	10.40	2.79	6.72
19R	2755	—	0.086	0.007	—	0.002	16.7	5.71	3.81	3.18	43.3	13.76	3.44	9.11
24R	2960	—	0.024	0.006	—	0.002	21.3	7.6	3.9	1.86	42.4	12.42	2.79	6.72
25R	2855	—	0.121	0.012	—	0.002	22.7	10.3	3.16	2.0	38.1	13.16	2.79	6.72
26R	2955	0.072	0.037	0.009	0.032	0.002	20.0	6.6	3.4	2.26	38.9	13.39	3.44	9.01
27R	2865	0.13	0.075	0.007	0.036	0.002	16.77	4.72	3.29	2.14	42.5	14.51	2.46	10.27

* Composite analyses.
† 15 min before to 15 min after end of flush.
‡ Numbers indicate individual heats, letters designate period of heat, and subscript 2 indicates the second of two samples taken.
§ 15 to 45 min after end of flush.

In general, then, it appears that the slag and bath temperatures take the following courses: Early in the flush the bath is cool; and, although the slag is exposed to the very hot flame, it is kept relatively cool by being mixed violently with the metal. As the vigorous action subsides, the slag picks up temperature rapidly and will frequently rise to over 2900°F. The temperature of the metal increases gradually from as low as 2400°F up to 2600° or 2700°. As soon as lime begins to come up, the slag will cool down again.

A few measurements in a relatively shallow-hearthed 250 ton furnace (35 in. maximum depth) showed that there was a much more rapid rise in bath temperature and generally there was a smaller difference between the slag and metal temperatures in the postflush period than was the case with the deep-hearthed 205 ton furnace (48 in.).

Table III. Comparison of Temperatures and Temperature Gradients of Bath and Slag in Postflush Period

Heat No.	Temperature, °F		Temperature Gradients, °F		Time and Period
	Slag	Metal	Slag	Metal	
			205 Ton Furnace		
353	2790	2550	60	—	10:50; Finish metal, 10:18; Finish flush, 11:00
893	2630	2450	60	0	8:43; Finish metal, 7:25; Finish flush, 8:40
	2730	2530	120	25	8:55
	2770	2540	60	10	9:15
	2810	2680	55	10	9:35
891	2950	2650	—	—	6:40; Finish metal, 5:56; Finish flush, 6:35
	2980	2630	70	30	6:50 (See Fig. 9)
682	2950	2550	—	—	3:15; Finish metal, 2:15; Finish flush, 3:00
	2840	2560	—	—	3:40
711	2640	2530	—	—	1:35; Finish metal, 12:30; Finish flush, 1:35
			250 Ton Furnace		
352	2900	2460	—	—	1:05; Finish metal, 12:17; Finish flush, 1:15
348	2860	2610	100	50	6:50; Finish metal, 6:05; Finish flush, 6:50
170	2790	2510	60	0	12:00; Finish metal, 11:15; Finish flush, 12:15
	2780	2560	—	—	12:15
	2900	2640	—	10	

The data and observations indicate that the temperatures and temperature gradients in the slag and metal in the flush and postflush periods are strongly influenced by physical conditions in the furnace. An ore charge which rises to the slag without reacting tends to cool the slag. A system in violent agitation will tend to keep the slag temperature down but will tend to bring the bath temperature up rapidly. All else being equal, it appears that a system having the larger hearth area per unit of metallics will tend to have the lower temperature differential.

Use of Existing Correlations

The first step in ascertaining whether or not a close approach to equilibrium is the dominant factor in determining the slag-metal distribution of phosphorus and manganese in the late and postflush periods is to make use of correlation methods which have been successful in treating other data that appear to represent equilibrium. Refining slags have shown themselves to be reasonably close to equilibrium,[1,3] for phosphorus and manganese 20 to 40 min after a major addition to the furnace. As a result, the postflush slag-metal systems in this study are correlated along with paired refining slags which were selected from the same heats and with refining slags from another study.

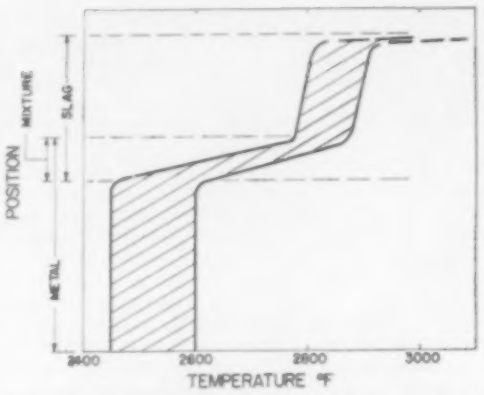

Fig. 2—Temperatures in slag and metal in postflush period are presented schematically.

Phosphorus: From equilibrium data, Winkler and Chipman[4] established the following equation and constant for the distribution of phosphorus between a bath of carbon-free iron and basic slags*

* Conventionally, CaO′ is available lime, ( ) designates slag components, and [ ] designates metal components.

$$2[\text{Pct P}]+5[\text{Pct O}]+4(\text{CaO}')=(4\text{CaO}\times\text{P}_2\text{O}_5) \quad [1]$$

$$\log K_p = \log \frac{(4\text{CaO}\times\text{P}_2\text{O}_5)}{[\text{Pct P}]^2[\text{Pct O}]^5(\text{CaO}')^4} = \frac{71{,}667}{T} - 28.73. \quad [2]$$

Eq. 1 and constant (Eq. 2) have been modified to apply to the basic open hearth system[5] as follows

$$2[\text{Pct P}] + 5(\text{FeO}) + 4(\text{CaO}')=(4\text{CaO}\times\text{P}_2\text{O}_5) \quad [3]$$

$$\log K'_p = \log \frac{(4\text{CaO}\times\text{P}_2\text{O}_5)}{[\text{Pct P}]^2(\text{FeO})^5(\text{CaO}')^4} = \frac{40{,}067}{T} - 15.06. \quad [4]$$

Herasymenko and Speight[6] have presented an ionic treatment of slags. Based on Winkler and Chipman's data, they established the following constant

$$\log K''_{P_2} = \log \frac{(\text{PO}_4^{'''})}{[\text{Pct P}]\ (\text{Fe}^{''})^{5/2}\ (\text{O}^{''})^{5/4}} \quad [5]$$

$$\log K''_{P_2} = 7(\text{Ca}^{''}) + \frac{14{,}660}{T} - 7.44. \quad [6]$$

The flush slag data from Table II are applied to Eqs. 4 and 6 with the results as shown in Figs. 4 and 5. Fig. 4 shows Winkler and Chipman's curves for 2910°F, extrapolated towards the acid side. If the assumptions of slag behavior inherent in the Winkler-Chipman treatment[4] are still valid in the vicinity of 2500°F, the phosphorus distribution ratio

$$R = \frac{(4\text{ CaO}\times\text{P}_2\text{O}_5)}{[\text{Pct P}]^2}$$

may be expected to be approximately 10⁶ times as great for a given slag at 2500°F (the average bath temperature in this study), as at 2910°F. Thus, in Fig. 4 if an equilibrium condition is being approached for the data plotted, it would appear that the apparent temperature for this equilibrium is nearer 2900° than 2500°F. It must be recognized that the

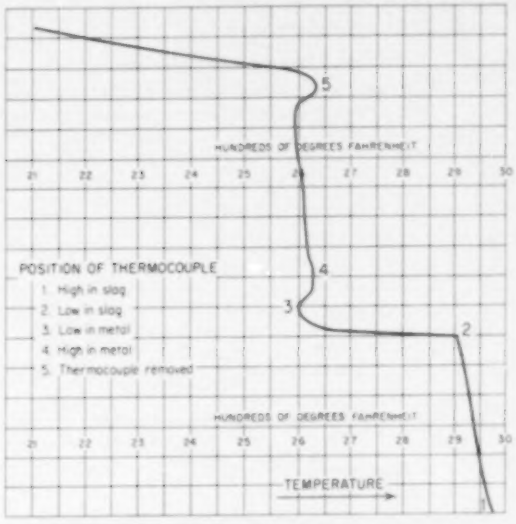
HUNDREDS OF DEGREES FAHRENHEIT
21 22 23 24 25 26 27 28 29 30
POSITION OF THERMOCOUPLE
1. High in slag
2. Low in slag
3. Low in metal
4. High in metal
5. Thermocouple removed
TEMPERATURE

Fig. 3—Temperature trace shown was taken as thermocouple was lowered through slag and moved in bath, 55 min after hot metal was added in 205 ton furnace, heat No. 891.

system is not in thermal equilibrium. These relatively acid slags, when plotted according to Eq. 6 on Herasymenko and Speight's[6] plot (Fig. 5), also indicate that it is possible to interpret the data as representing equilibrium at approximately 2900°F. From the follow-up study of slag and metal temperatures on this furnace, it appears that it is more pertinent to think of the slag temperature than the metal temperature. Plotting these same data as log K'_p (Eq. 4) vs $1/T$ also tends to verify the viewpoint that there is an apparent temperature of equilibrium which lies very close to the approximate slag temperature. The three methods of treating the data are all related and, consequently, really give only a single comparison with established equilibrium conditions from other studies.

It is considered probable that the distribution of phosphorus between slag and metal is very close to equilibrium, based on the estimated average slag temperature in the late and postflush periods because:

1—With the exception of the effect of metal temperature, the data fall quite consistently on established correlation plots.

2—The *apparent temperature of equilibrium* from the methods of correlation is very close to the temperature of the body of the slag (approximately 2900°F) as measured on several very similar heats in the early postflush period.

3—The manganese and phosphorus contents of the metal reach low levels late in the flush period. Subsequently, levels of these components in the bath gradually increase as slag oxidation drops due to carbon removal (Fig. 1). It is felt that this reversal of direction of net movement indicates strongly that equilibrium has been reached. The exchange of these components from the slag to metal could well be controlled by the slag temperature rather than the bath temperature, because it has been found that the level of oxidation of the slag rather than the level of bath oxidation is of paramount importance in the refining reactions.[1] Also, phosphorus and manganese are relatively high in the slag and low in the bath. It must be noted that this is the reverse of the condition early in the flush when the metal is high in these two components while the slag is low.

4—The transfer of phosphorus from slag to metal was shown to take about 15 min to reach equilibrium in an induction furnace.[8] The violent agitation of metal and slag in the flush period in the open hearth probably ensures as good or better mixing. Early in the flush in the open hearth furnace, the transfer of phosphorus is from metal to slag but later it apparently reverses and takes place from slag to metal as was the case in the induction furnace work. The smooth and consistent increase and then decrease of phosphorus in the slag accompanied by a corresponding decrease and then increase in phosphorus content of the metal indicates that the rate of approach to equilibrium is fully as fast in the open hearth as in the induction furnace.

5—Conditions during the refining period are not greatly dissimilar to those during the late and postflush period. It has been demonstrated that refining slags approach equilibrium with regard to phosphorus quite closely.[1] Major differences are that during the flush period the carbon content of the metal is high rather than low and the slag FeO is changing rapidly rather than slowly.

Manganese: Darken and Larsen[9] have correlated the manganese distribution between slag and metal in a large number of basic open hearth heats by the

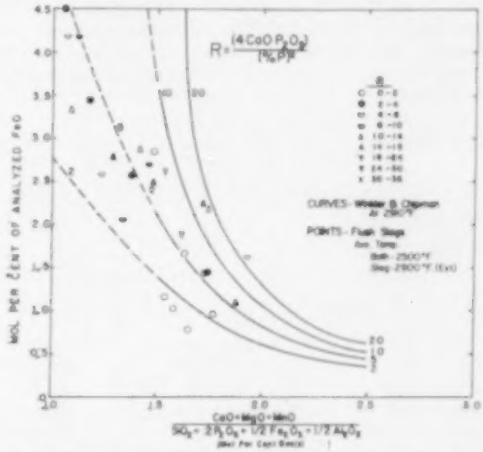

Fig. 4—Dephosphorization ratios in postflush period are shown. Curves are from data of Winkler and Chipman.[5]

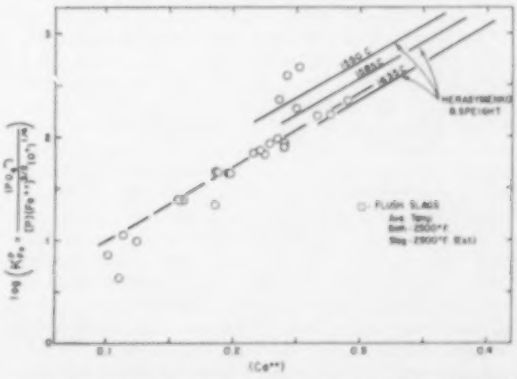

Fig. 5—Postflush dephosphorization was correlated by Herasymenko's[6] method.

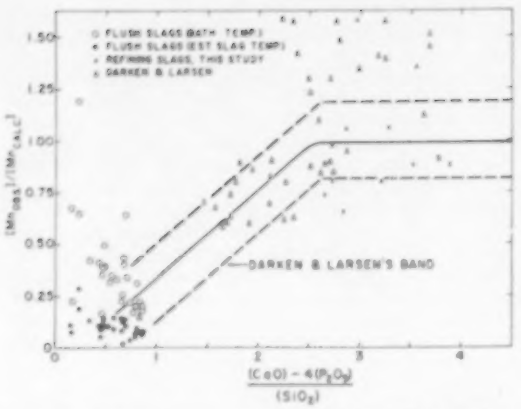

Fig. 6—Postflush manganese chemistry was correlated by Darken and Larsen's[5] method, and selected data from Darken and Larsen were used.

method shown in Fig. 6. The method of correlation is that proposed by them but the points contained in the figure are calculated only from data in ref. 2 for which slag temperatures were available. As a consequence, Fig. 6 is not a duplication of Fig. 1, ref. 2. It is quite evident that the data from operating furnaces show a rather large spread which may result from lack of precision in chemical analyses, from lack of adequate data on temperatures, and perhaps, in some cases, from lack of a stabilized system such as a raw slag. [Pct Mn] obtained for Fig. 6 was calculated as follows

$$[\text{Pct Mn}]_{\text{calculated}} = \frac{(\text{MnO})}{(\text{FeO}_t)\ K_{\text{Mn}_{1600}}}. \qquad [7]$$

(FeO_t) is the mol fraction of ferrous oxide calculated from the total oxygen combined with iron.

$K_{\text{Mn}_{1600}}$ is the constant for the reaction

$$[\text{Mn}] + (\text{FeO}_t) = (\text{MnO}) + [\text{Fe}] \qquad [8]$$

according to Körber and Oelsen[4] where

$$K_{\text{Mn}} = \frac{(\text{MnO})}{(\text{FeO}_t)\ [\text{Pct Mn}]}. \qquad [9]$$

Körber and Oelsen established that in simple oxide slags the activities of (MnO) and (FeO_t) are proportional to their molar concentrations in the slag and that the activity of manganese in the metal is proportional to [Pct Mn]. The activity of iron closely approaches one.

The postflush data (Table II) are entered in Fig. 6 with $[\text{Pct Mn}]_{\text{observed}}/[\text{Pct Mn}]_{\text{calculated}}$ determined from $K_{\text{Mn}_{1600}}$ at the respective bath temperatures. The same data are recalculated using the constant for the *approximate slag temperature*. These temperatures are obtained by adding 400°F to the bath temperature in each case. This method of correlation is not considered sensitive enough to show how closely manganese distribution between slag and metal approaches equilibrium. In addition, the flush slags reported here fall at the edge of Darken and Larsen's range of basicity. All that can be said about the flush data being considered is that they correlate reasonably well with refining slags which probably closely approach equilibrium with the bath with respect to manganese. Points based on the estimated slag temperatures show less spread and fall more closely within the range established by Darken and Larsen's points than if they were based on the measured bath temperatures.

Proposed Manganese Correlation

Since Darken and Larsen[5] proposed their correlation (Fig. 5), much more information has been accumulated on the behavior of basic open hearth-type slags. Taylor and Chipman[6] have established the activity surface of (Fe_tO)† over a portion of the

†Fe_tO is the total iron of the slag calculated to FeO (Fe_tO = FeO + $2Fe_2O_3$). FeO_t is the total oxygen combined with iron calculated to FeO (FeO_t = FeO + $3Fe_2O_3$).

(CaO + MgO)-SiO_2-Fe_tO system. In addition, Chipman, Gero, and Winkler[7] have redetermined the value of K_{Mn} (Eqs. 8 and 9) as follows for steelmaking temperatures

$$\log K_{\text{Mn}_{1600}} = \frac{6440}{T} - 2.95. \qquad [10]$$

Eq. 9 is more properly written

$$K_{\text{Mn}} = \frac{a\ (\text{MnO}) \times a\ [\text{Fe}]}{a\ (\text{Fe}_t\text{O})\ a\ [\text{Mn}]}. \qquad [11]$$

The equation can be simplified and altered as shown with the following modifications

$$K_{\text{Mn}} = \frac{(\text{MnO})\ \gamma_{(\text{MnO})}}{a\ (\text{Fe}_t\text{O})\ [\text{Pct Mn}]}. \qquad [12]$$

a [Fe] approaches 1, a [Mn] = [Pct Mn], and a (MnO) = (MnO) $\gamma_{(\text{MnO})}$. Knowledge of a slag-metal system at equilibrium permits the calculation of γ(MnO)

$$\gamma\ (\text{MnO}) = \frac{a_{(\text{Fe}_t\text{O})}\ K_{\text{Mn}}\ [\text{Pct Mn}]_{\text{observed}}}{(\text{MnO})}. \qquad [13]$$

In order to use Eq. 13, the quantity $a_{(Fe_tO)}$ is required for each slag. This was obtained from Fig. 7 which is based primarily on Taylor and Chipman's[6] data and on Winkler and Chipman's[8] results. The Fe_tO-SiO_2 side was recalculated from Schuhmann and Ensio's[9] and Gokcen and Chipman's[10] data. Activity of Fe_tO on the CaO-Fe_tO side was calculated from Taylor and Chipman's[6] curves extrapolated to this side. All of these sources were interrelated. Publication elsewhere of the results is expected soon.

Fig. 8 shows data from basic open hearth-type slags calculated according to Eq. 13 and plotted against slag basicity. The points from Winkler and

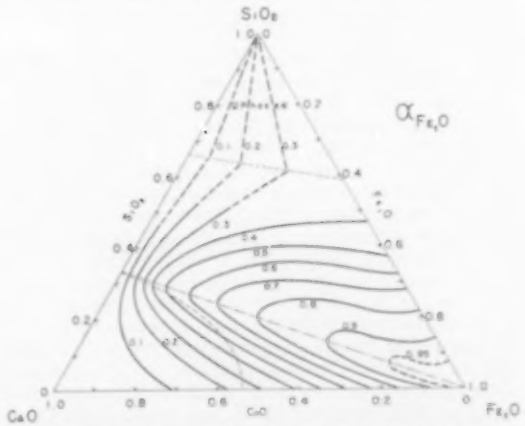

Fig. 7—Activity of Fe_tO in CaO (bases)-SiO_2 (acids)-Fe_tO system is shown, based on data of Taylor and Chipman,[6] Winkler and Chipman,[8] Schuhmann and Ensio,[9] and Gokcen and Chipman.[10]

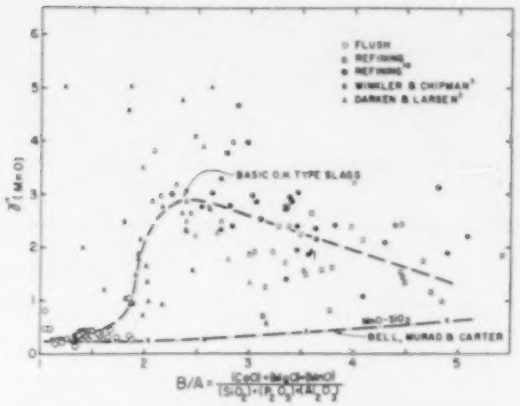

Fig. 8—Data points and curves illustrate effect of basicity on $\gamma_{(MnO)}$ in basic open hearth-type slags.

Chipman[9] are only those which fall in the range of usual basic open hearth slag analyses. The selected points calculated from Darken and Larsen's data[7] which appear in Fig. 6 are also shown here. Furnace data from Tenenbaum‡ are included as well.§

‡ Private communication. Data are used in Figs. 17 through 21, ref. 1.

§ Darken and Larsen's data are calculated to the present correlation by means of the following equation which can be derived from Eqs. 9 and 13

$$\gamma_{(MnO)} = \left[\frac{[\text{Pct Mn}]_{observed}}{[\text{Pct Mn}]_{calculated}}\right] \times \frac{a_{(Fe_tO)}K_{Mn_{(COW)}}}{(Fe_tO)\ K_{Mn_{(EO)}}} \quad [14]$$

The scatter in the data is very large, both for the operating furnaces and the laboratory studies. This may result in part from a lack of high precision in the analytical procedures such as in determining low manganese contents in the metal bath. It also stems from the fact that this correlation ignores the fact that the activity coefficient of MnO will vary with basicity (see Fig. 9). Thus, as will be shown later, some spread in the data in Fig. 8 would result even if there was no experimental error. The data from these two papers are for the most part from slags carrying 0.01 to 0.03 mol fraction of MnO. The $\gamma_{(MnO)}$ curve is brought down sharply from 3 to less than 1 in crossing the basicity line of 2 in the direction of higher to lower basicity. The change in the behavior of (MnO) in this region of the dicalcium silicate-(Fe,O) pseudo-binary is similar to what would be expected for lime. However, the change is somewhat less marked, probably because lime is considered to be a much stronger base than is MnO.

Data for the MnO-SiO_2 system[10] are also shown in Fig. 8. It is probable that with a basicity ratio of less than 1, MnO will function as a reasonably strong base. From that point to silica saturation, (MnO) = 0.44, $\gamma_{(MnO)}$ equals about 0.25. Using this as a guide to the behavior of MnO in the somewhat acid range, the curve representing $\gamma_{(MnO)}$ is brought in tangent to the open hearth line below a basicity of one.

The heavy line from Fig. 6 can be calculated to fall on Fig. 8 by Eq. 14. It converts as a band (not shown) which follows the dashed line. The breadth of the band is a result of the variation of $\gamma_{(P_2O_5)}$ (Fig. 10) along each line of constant basicity in the ternary composition system. $\gamma_{(P_2O_5)}$ is found by dividing the real value of $a_{(P_2O_5)}$ by $N_{(P_2O_5)}$. The band is very narrow at B/A equals one; it spreads over a range of two units in the range of B/A equals two and narrows again for higher basicities.

The points for the slag-metal systems during and after the flush period are grouped closely about the location of the line from Bell, Murad, and Carter's[10] data and the curve sketched in Fig. 8. These points were computed using the estimated slag temperatures. The same data calculated using actual bath temperatures have values of $\gamma_{(MnO)}$ approximately three times those shown. If the argument that the slag temperatures are controlling in the manganese reaction is accepted for the same general reasons as proposed in the case of phosphorus, then these data are reasonably consistent with other data which are at or very close to equilibrium. It thus appears that manganese distribution in the flush and postflush periods may be rather close to equilibrium which is controlled by the slag temperature.

Discussion

The study of conditions existing during the flush and postflush periods of basic open hearth operations has focused attention on the generally known fact that the slag is not in thermal equilibrium with the bath. In the furnace studied, the slag is apparently much hotter than the metal by a matter of 300° to 400°F. In other furnaces the differential may be somewhat smaller but the slag temperature is still close to 2900°F. A large number of temperature measurements in the furnace showed very little difference in the bath temperatures from heat to heat during the period discussed. This is in spite of widely differing types of scrap charged, different melt-down conditions, and different charge oxides.

The rapid carbon drop during the postflush period indicates quite clearly that the slag-metal system is far from equilibrium with respect to carbon.

During the early period of flush, it appears that silicon, manganese, and phosphorus are removed somewhat in the order of the stability of their oxides. Because of the strong oxidizing action of the charge ore, mass transfer of these elements from metal to slag is very rapid and the periods for removal of the elements overlap.

During the flush the strongly oxidizing ore drives manganese and phosphorus into the slag. As a result, in the postflush period when the oxygen level of the slag is dropping and the temperature is rising, there is a slight reversion of these two elements from the slag to the metal. At this point, the system appears to be quite close to chemical equilibrium and may be able to stay so because of the small mass transfer of phosphorus and manganese which is necessary to adjust for the continuing decrease in slag oxidation. As pointed out previously, there are a number of reasons why the system may be close to equilibrium with regard to manganese and phosphorus in spite of the fact that it is not with respect to temperature, carbon, and oxygen. With the small net transfer of manganese and phosphorus being from slag to metal at this period, there seem to be reasonable arguments for the case that slag temperatures should be controlling. Although large temperature gradients are known to exist between slag and metal, the *apparent equilibrium temperature* obtained from the phosphorus data appears to be very close to the slag temperature. Calculations for the manganese distribution tend to bear out the conclusion from the phosphorus data.

A little data thus far collected indicate that the conditions described herein for phosphorus and man-

ganese during and after the flush may also apply shortly after the addition of hot metal in scrap heats.

A further commentary on Fig. 8 may be of interest. The various points for the experimental flush, postflush, and finishing slags are shown on a portion of the composition triangle in Fig. 9 and lines of constant $\gamma_{(MnO)}$ are drawn. Some of the spread of the points in Fig. 8 results from the iso-$\gamma_{(MnO)}$ curves not being parallel to the lines of constant basicity, Fig. 9. It must be recognized that this plot assumes that the points from the postflush slag data which fall along the line of $\gamma_{(MnO)} = 0.3$ are at equilibrium.

The isoactivity coefficient lines in Fig. 9 demonstrate that the tendency toward association of CaO with SiO_2 dominates the system. Similarly to (Fe_tO), Fig. 10, (MnO) has the area of the highest activity coefficient in the region between the binary lines connecting $3CaO \cdot SiO_2$ and $3CaO \cdot 2SiO_2$ with the Fe_tO apex. MnO shows its basic behavior on the acid side because the activity coefficient surface drops rapidly as the line of $B/A = 1$ is approached. In comparing the activity coefficients of MnO and Fe_tO in this composition range, it is noted that the maximum observed value for $\gamma_{(MnO)}$ is approximately three, while $\gamma_{(Fe_tO)}$ is over four. The displacement of the ridge for (MnO) toward the bases corner as compared to that for (Fe_tO) tends to indicate that MnO is a stronger base than is Fe_tO.

Conditions of violent mixing of slag and metal and the presence of undigested lumps of lime in the slag which occur during the lime boil thus far have prevented a study of conditions with regard to manganese and phosphorus during this period.

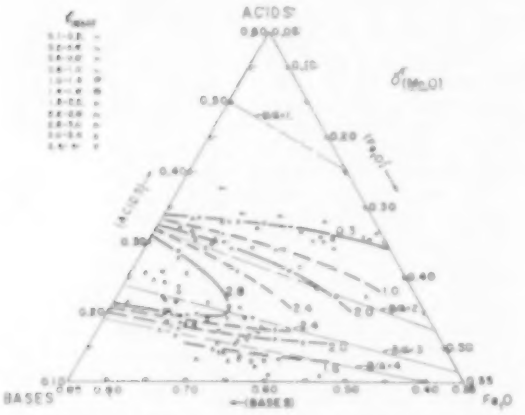

Fig. 9—Estimated iso-$\gamma_{(MnO)}$ curves are shown as a function of composition in basic open hearth-type slags in a portion of bases-acids-Fe_tO system.

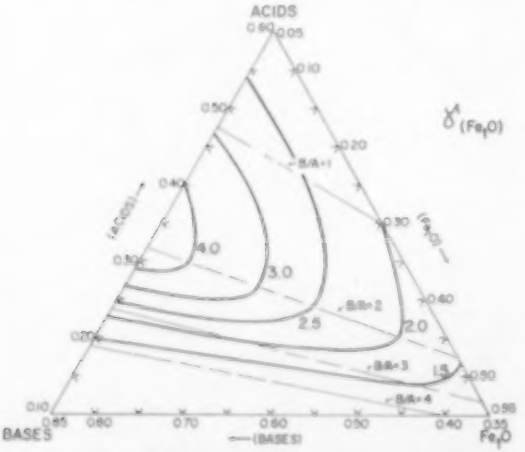

Fig. 10—Isoactivity coefficient lines for Fe_tO were calculated from data given in Fig. 7.

Summary

Data are presented which tend to indicate that phosphorus and manganese distributions between slag and metal closely approach equilibrium toward the end of flush and just after flush in the basic open hearth furnace. The apparent temperature for these equilibria appears to be much closer to the average slag temperature than to the bath temperature. At this period in the heat, a temperature difference of as high as 450°F may exist between the two phases. This difference is strongly dependent on the furnace system itself and the particular conditions prevailing in each heat.

The effect of slag composition on the activity coefficient of MnO in the slag is estimated.

Acknowledgments

The authors wish to thank M. Holowaty and E. Travis for their efforts during the test work, and the members of Open Hearth Dept., Inland Steel Co., for their excellent cooperation in the experimental program. They wish to express their appreciation to the Inland Steel Co. for permission to use and to publish the open hearth data contained herein.

Discussion of this paper, if any, will appear in JOURNAL OF METALS, November 1956, and in AIME Metals Branch *Transactions*, Vol. **206**, 1956.

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aime NEWS

C. F. Kettering To Receive Hoover Medal

The Hoover Medal for 1955 will be presented to Charles F. Kettering during the Diamond Jubilee Annual Meeting of the American Society of Mechanical Engineers in Chicago, Nov. 13 to 18, 1955. The medal is given for distinguished public service by an engineer. Dr. Kettering graduated as a mechanical engineer from Ohio State and has honorary degrees from 29 other institutions. He served for 27 years as vice-president of General Motors Corp. and general manager of its research laboratories. A large number of automotive inventions are attributable to him. He is now retired and lives in Dayton.

Pittsburgh Extends New Building Offer

The Board of Trustees of the Pittsburgh Engineering Trust has voted to extend its offer of $1.5 million to help finance a new engineering building to Apr. 1, 1956. The expiration date had previously been Sept. 1, 1955. As soon as all five of the societies concerned have ratified the new plan for a committee of 15 to study all factors in the situation and make a recommendation, following an objective report by an outside firm of professional consultants, it is hoped that the five boards of direction may unanimously accept whatever recommendation is made. The matter should then be settled soon after the turn of the year.

Pittsburgh NOHC To Have 10th Anniversary

The AIME-NOHC Pittsburgh Section will hold its 10th Anniversary Meeting, Friday, Nov. 4, 1955, at the William Penn Hotel. It will be the traditional Off-the-Record meeting.

Simultaneous meetings will be held during the morning on Acid Open Hearth and Refractories and Combustion. Included under Acid Open Hearth are papers on *Vacuum Casting of Large Forging Ingots* and *Aluminum in Acid Open Hearth Steels*. The Refractories and Combustion session will cover combustion, masonry, and operations practice in all their phases.

In the afternoon the Metallurgy and Operating sessions have papers on *Desulphurization of Hot Metal for the Open Hearth, Desiliconization of Hot Metal for the Open Hearth, The Control of Residual Metallic Aluminum in the Production of Deep Drawn Steels*, and *The Benefits to be Derived Through the Use of Open Hearth Bath Temperature Controls*. In addition motion pictures will be shown on the control of ingot surface quality.

S. J. Dougherty, Weirton Steel Co., is Chairman of the Anniversary Meeting and E. B. Snyder, Wheeling Steel Corp., is Program Chairman.

IMD Plans Seminars During March 1956

The Niagara Frontier Section in conjunction with the IMD is arranging to hold a three-day Conference on Reactive Metals on Mar. 19, 20, and 21, 1956. Subjects of the conference will include the research and practical aspects of the metallurgy of titanium, zirconium, molybdenum, rhenium, vanadium, columbium and hafnium. There will be plant trips to plants in the area engaged in research and fabrication of these metals. The arrangements are in the hands of R. W. Fountain, C. W. Hart, G. L. Cox, and Loren Smith.

About one week later on March 29 and 30, the Metals Branch, Southern California Section, with the IMD, will hold a Symposium on Physical Metallurgy of Titanium at the University of California at Los Angeles. The program for this meeting tentatively will include papers describing both titanium technology and recent research on the physical metallurgy of titanium. The Committee for Arrangements consists of Leonard Jaffe, Chairman; Pol Duwez, Jim Long, and Jack Taylor.

A. Buzzalini Chosen Mining Branch Secretary

Arnold Buzzalini has been engaged as the new Secretary for the Mining Branch of the AIME. Mr. Buzzalini most recently has been manager and

ARNOLD BUZZALINI

chief geologist of the uranium div., Pubco Development Inc., Albuquerque, N. M. He has also been secretary of the New Mexico Geological Society and was chairman of the committee for its ninth annual meeting last May in Gallup, N. M.

Arnold, or "Buzz" as he is sometimes called, comes from a mining family. He went to Syracuse University for his geology training, later doing graduate work at Johns Hopkins and Penn State. His college career was interrupted by a three-year stretch in the Air Force, principally in Alaska and the Yukon.

After completing his formal schooling he spent a year in geological work in the East and then went with Phillips Petroleum Co., Bartlesville, Okla., in 1951 as a staff geologist on exploration and development. Last year he joined the company's strategic minerals div. and a few months ago joined the staff of his present employer. He has visited mines in the West, extending from Alaska and the Yukon to Pachuca in Mexico.

Processed for M

ceiving his Ph.D. in chemistry in

Personals

W. C. RUECKEL

W. C. Rueckel has accepted the position of vice president and general manager of the engineering construction div., Koppers Co., Pittsburgh. He had been vice president, Kaiser Engineers, Oakland, Calif.

Paul Schwarzkopf was elected president of American Electro Metal Corp. of Delaware.

John E. DeMoss has joined the General Electric Co., West Lynn, Mass. Mr. DeMoss is supervisor of metallography and high temperature testing in the small aircraft engine dept. laboratory. He had been chief metallurgist of the powder metal parts div., Keystone Carbon Co., St. Marys, Pa.

Sheldon Weinig recently received his doctor of engineering science degree and is now assistant professor of metallurgy, College of Engineering, New York University. He had been a lecturer in physical metallurgy at Columbia University, New York.

George J. Fischer, director of the dept. of metallurgy, Sam Tour & Co., Inc., New York, has resigned to join the staff of the Polytechnic Institute of Brooklyn.

J. P. Warner is economist at the chemical metallurgical div., Sherritt-Gordon Mines Ltd., Fort Saskatchewan, Alberta. He had been on the staff of the University of British Columbia.

Erwin O. Deimel has accepted the position of product design engineer, General Electric Co., light military electronic equipment dept., Utica, N. Y. He had been associated with the Utica Drop Forge & Tool Corp., as senior research metallurgist.

C. William Conn, manager, electric and open hearth furnaces, Ford Motor Co., Dearborn, has been promoted to production manager of the steel div.

Ursula E. Wolff has joined the General Electric Co., lamp wire and phosphorus dept., Cleveland.

Robert S. Hogue, National Carbon Co., has been transferred to Pittsburgh from New York.

Harry L. Anderson, Jr., Republic Steel Corp., Youngstown, is superintendent, masonry dept.

Richard F. Gieselman has joined the Eureka Williams Co., div. of Henney Motor Co., Inc., Bloomington, Ill., as design engineer in ordnance development. Mr. Gieselman had been associated with the meadows div., Thor Corp.

Emil A. Kovacevich, John P. Page, and **William C. Thurber** have been appointed to the staff of the Oak Ridge National Laboratory, Oak Ridge.

John F. Elliott, former assistant superintendent, Inland Steel Co., East Chicago, Ind., has joined Massachusetts Institute of Technology, as an associate professor.

Gordon W. Johnson is now project supervisor for Hunter Engineering Co., Riverside, Calif. He had been assistant director of production research, Vanadium Corp. of America, Cambridge, Ohio.

W. T. Hudson has resigned as division manager of Ralph Fair, Inc., and will reside in Denver to engage in private business.

William Justin Kroll has been selected to receive the Albert Sauveur Achievement Award presented by ASM. **Alfred L. Boegehold,** head of the metallurgy dept., research laboratories div., General Motors Corp., is 1955 recipient of the ASM Gold Medal.

W. John King is assistant plant superintendent, Kaiser Aluminum & Chemical Corp., Newark, Ohio. Mr. King was formerly plant manager for Knoxville Iron Co., Knoxville, Tenn.

Philip R. White is a member of the technical staff, Bell Telephone Laboratories, Inc., Murray Hill, N. J. Mr. White had been a member of the research laboratory div., General Motors Technology Center, Warren, Mich.

Joseph Burr Tyrrell, director, Kirkland Lake Gold Mining Co., Ltd., Toronto, has been awarded Honorary Membership by the Institution of Mining and Metallurgy, London. Dr. Tyrrell was born in Canada in November 1858.

A. E. Lee, Jr., American Metal Co., Ltd., is now located in New York. He had been with the Blackwell Zinc Co., Inc., Blackwell, Okla.

C. S. Wiedman, General Electric Co., Detroit, Carboloy dept., has been appointed manager of carbide products development engineering. Prior to the new appointment, Mr. Wiedman had been product and process development engineer.

John E. Hatch has been transferred by the Aluminum Co. of America from New Kensington, Pa., to Davenport, Iowa. He will be assistant chief metallurgist.

R. Ward, General Electric Co., has been transferred to Shelbyville, Ind. from Schenectady. The company has moved its industrial heating dept. to Shelbyville.

E. M. Fitchett is now chief chemist for the Ray-O-Vac Co., Madison, Wis. He had been associated with the Portland American Metallic Chemicals Corp.

George M. Baumann, American Smelting & Refining Co., has been transferred from Whiting, Ind. to New York. Mr. Baumann is in the Federated Metals div.

R. Earl Penrod has retired as chief metallurgist of the Johnstown plant of Bethlehem Steel Co. **E. H. Hollenback,** former assistant chief metallurgist, will succeed Mr. Penrod.

John Glasgow, formerly assistant to the superintendent, open hearth dept., Jones & Laughlin Steel Corp., has been promoted to superintendent of the open hearth dept.

Bernard de la Brunière, technical advisor and U. S. representative for Compagnie de Pont-a-Mousson, has returned to France for consultation on new technical developments. After visiting company plants in Pont-a-Mousson, Nancy, and Nelun he will return to the U. S. next March.

B. DE LA BRUNIERE

Proposed for Membership
— Metals Branch AIME—

Total AIME membership on Aug. 31, 1955 was 23,194, in addition 1903 Student Associates were enrolled.

ADMISSIONS COMMITTEE

P. D. Wilson, Chairman; F. A. Ayer, Vice-Chairman; A. C. Brinker, R. H. Dickson, T. D. Jones, F. T. Hanson, Sidney Rolle, O. B. J. Fraser, F. T. Sisco, Frank T. Weems, R. L. Ziegfield, R. B. Caples, F. W. McQuiston, Jr., A. R. Lytle, H. R. Wheeler, L. P. Warriner, J. H. Scaff.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

In the following list C/S means change of status; R, reinstatement; M, member; J, Junior Member; A, Associate Member; S, Student Associate.

Connecticut

Fairfield—Cleveland, George W. (J)
Hartford—Roberts, Kenneth D. (M) (R. C/S—J-M)
New Canaan—Caron, Peter E. (A)

Illinois

Crete—Coddington, William H. (A)
Marengo—Garvin, Stefan J. (M) (C/S—J-M)

Montana

Columbia Falls—Hook, Albert (A)
Columbia Falls—Taylor, Charles E. (M)

New Jersey

Whippany—Starr, C. Dean (M) (R. C/S—S-M)

New York

Bayside—Brandt, Helmut H. (A)
Pelham Manor—Prince, Charles H. (J)
Rochester—Ramsay, Glendon J. (A)

Ohio

Steubenville—Thompson, Lawrence L. (M)

Pennsylvania

Bethlehem—Stahl, Charles W. (M)
Pittsburgh—Mullins, William W. (J)

Obituaries

Edwin Letts Oliver

An Appreciation by
T. B. Counselman

On August 28th the American Institute of Mining and Metallurgical Engineers lost one of its most distinguished members, Edwin Letts Oliver, who died suddenly at his summer home in Lake Tahoe.

Mr. Oliver was a Life Member of AIME, having joined in 1902, and was a Member of the Legion of Honor Class of 1952. He was a past Chairman of the San Francisco Section, 1939, was elected a Director of the Institute in 1953, and was the recipient of the James Douglas Gold Medal for distinguished achievement in nonferrous metallurgy in 1955.

Edwin Letts Oliver was born in 1878, graduated from the College of Mines of the University of California in 1900, and received the honorary degree of LL. D. from that same university in 1945. His career in metallurgy was long and distinguished. Soon after graduation, he joined North Star Mines in Grass Valley, Calif., and became involved in the improvement of gold recovery and decreasing of tailings losses. This led to the development of the

EDWIN LETTS OLIVER

Oliver continuous filter, the first successful unit of its type.

After conducting his filter business at night and on weekends for two years, while continuing as metallurgist of the North Star Mines, Mr. Oliver finally resigned from the latter position and in 1910 formed the Oliver Continuous Filter Co., with offices in San Francisco, to market his inventions.

Through the years, various other types of filters and auxiliary equipment were invented or acquired and in 1925 the Oliver United Filters Inc. was formed, with subsidiary companies in England and France. Mr. Oliver continued as president of this company until Dec. 31, 1954, when it was merged with The Dorr Co. to form Dorr-Oliver Inc., of which he became one of the two founder-chairmen.

Mr. Oliver was active in many phases of metallurgy besides the operation of the company he founded. In his early days, he designed and built several cyanide plants in California and operated a number of gold mines and mills. He was one of the founders of the famous Idaho-Maryland mine, for many years one of the largest of the U. S. gold producers.

Besides his active interest in AIME, Mr. Oliver was a member of the Mining and Metallurgical Society of America; Chemical, Metallurgical, and Mining Society of South Africa; Society of the Golden Bear; Sigma Xi; Delta Upsilon; and Sigma Gamma Upsilon, as well as the Engineers' Club and Bohemian Club, both of San Francisco.

Mr. Oliver is survived by his widow, the former Minnie Griffin Walker, to whom he was married in 1905; two sons, William L. and Edwin Letts, Jr.; and two daughters, Mrs. Fred L. Greenlee and Mrs. Adrian Shannon.

Gustav A. Lillieqvist (Member 1944), research director for American Steel Foundries, Chicago, died of a heart attack on May 31.

Mr. Lillieqvist attended Polytechnical Institute of Zurich and the University of Berne, Switzerland, receiving his Ph.D. in chemistry in 1924 from the latter institution. He was born in London, England, coming to the U. S. in 1924. In the same year he was hired as a chemist at the Granite City, Ill. Works of American Steel Foundries. In 1927 he was transferred to the research laboratory in East Chicago, Ind. He served there until 1941 when he was appointed assistant works manager and chief metallurgist of the Indiana Harbor Works. In 1943 he became research director for the company.

Mr. Lillieqvist had been very active in the metallurgy of cast steel and devised numerous alloys and deoxidation practices. In 1933 he was named recipient of the AIME R. W. Hunt Award.

An Appreciation of
Carl Albert Heberlein

By Max Heberlein

Carl Albert Heberlein passed away unexpectedly at the home of his son in Bronxville, N. Y., on May 6, 1955.

Born on Jan. 7, 1870 in Braubach, Germany, he received his chemical education at the University of Bonn and came to this country in the early nineties. He spent a number of years first as prospector and then as assayer and plant operator in Colorado, Utah, Idaho, and Washington in copper and lead smelters and refineries. After the turn of the century his duties took him to Chiuhuahua, Torreon, and Mapimi, Mexico, where he

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devoted himself exclusively to the smelting and refining of lead and the recovery of the separated impurities. He was the first operator of a Harris lead refinery in this country in the early twenties and consulted in matters relative to this process in the years following. During the decade before World War II his duties took him to Chile, Venezuela, and across the Pacific to Mount Elliot, Australia.

After serving the federal government as consultant during World War II he retired from active metallurgy and lived with his son in Bronxville, N. Y. ever since.

His diversified metallurgical experience was the reason that he counted such metallurgists as Messrs. Klepetko, Anton Eilers, Rath, as his closest friends from the good old rough nineties. On the other hand he possessed an exceptional talent for telling interesting Wild West stories of some of his early friends, such as Charley Russell, Buffalo Bill and other well known personalities of bygone days.

It is needless to say that he always found an attentive audience at Headquarters. Whoever knew him more intimately will regret immensely that Carl Albert Heberlein has gone, never to return again to the AIME lobby to recount some events of 50 to 60 years ago.

Stephen M. Swain (Member 1944) has died. Mr. Swain had been vice president in charge of research, North American Refractories Co., Cleveland. Born at Hornell, N. Y., he graduated from Alfred University in 1925. For four years he was on the staff of Mellon Institute. Mr. Swain joined North American in 1934.

Rufus E. Zimmerman (Member 1920), retired vice president and chairman of the research policy committee, U. S. Steel Co. died on June 21.

Dr. Zimmerman was born in Mount Pleasant, Pa., Nov. 26, 1886. He was graduated from Franklin and Marshall College in 1908 with a Ph.B. He continued his education at Massachusetts Institute of Technology, receiving his S.B. in 1911, and remained as an instructor in physical chemistry.

Dr. Zimmerman first became associated with steel research in 1914 when he joined the American Sheet & Tin Plate Co., which later became a part of U. S. Steel. In 1915 he was made assistant director of research laboratory of American Sheet and was made acting director of research in 1917. He became director of research in 1919 and assistant to vice president in 1922, a position he held until 1932. In that year he transferred to New York as assistant to president of U. S. Steel Corp., and was named vice president in 1933.

During the first world war Dr. Zimmerman was consulting chemical engineer for the U. S. Bureau of Mines.

Necrology

Date Elected	Name	Date of Death
1908	Charles P. Berkey	Aug. 22, 1955
1934	H. J. French	Aug. 17, 1955
1934	J. O. Greenan	July 23, 1955
1918	Hugo E. Hanser	July 14, 1955
1945	Carleton C. Hascall	June 2, 1955
1952	John D. Hysong	July 23, 1955
1915	Estey A. Julian	Aug. 2, 1955
1937	Frank L. Kennicott	June 29, 1955
1920	Lewis P. Larsen	July 14, 1955
1934	Donal F. McCarthy	May 10, 1955
1902	Edwin Letts Oliver	Aug. 28, 1955
1891	Frederick D. Power	June 28, 1955
1914	Gar A. Roush	Aug. 16, 1955
1946	Everett S. Shaw	June 30, 1955
1929	Robert B. Campbell	Aug. 16, 1955

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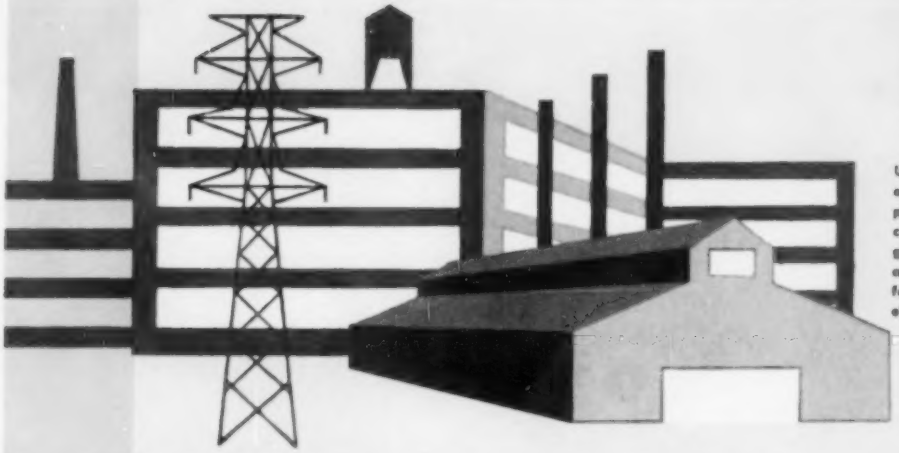

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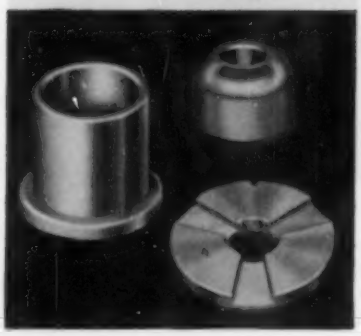

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